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INVESTIGATION OF PROBLEMS ASSOCIATED WITH SOLID ENCAPSULATION OF HIGH VOLTAGE ELECTRONIC ASSEMBLIES

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October 15, 1974

D. C. Teachers College
Miner Building
2535 Georgia Ave., N.W.
Washington, D.C. 12009

RE: Grant # NGR 09-053-003

Dear Mr. Westrom:

Due to the recent severe illness of my husband, I shall indeed take advantage of the fact that the six months status report can be concise and informal. Three projects have been worked on during the first six months of the grant period which are dicussed in some detail on the following pages. Some, but not all, of the data is presented in graph or calendar form. However, formalities such as Tables of Content and lists of references have been omitted at this writing.

I would like to take this opportunity to thank you for your continued guidance, advice and helpfulness with this project.

Yours sincerely,

Renate S. Bever

Renate S. Bever
Principal Investigator

I

ABSTRACT:

Results for the permeation constant of air through Shell Epon 828 Epoxy. Emerson & Cuming Stycast 3050 Epoxy, Thiokol Solithane and Dow-corning Sylguard 185 are presented at ambient room temperature.

Adhesive strengths were measured by the ASTM-1002 lap shear method. Epon 828 Epoxy was used as adhesive and various surface preparations of the adherends were tried on glass-epoxy circuit-board and on solder, electroplated on beryllium copper. Minimum estimates of adhesive strengths are given for procelain and ferrite.

The behavior of the Reynolds, series 600, high voltage connectors, filled at various pressures was studied, with 3 kilovolts applied. It was found that outgased connectors have zero or very low noise count above and below the corona region but do have some noise counts in the corona region. The Reynolds connectors do indeed suppress catastrophic breakdown when filled with gas at corona pressures even after numerous openings and closings.

I CONTINUATION OF THE STUDY OF PERMEATION OF AIR THROUGH SPACE GRADE INSULATIONS:

In an earlier report[(1)] the method, theory and some preliminary results for permeation of air through encapsulating polymers were presented. Enough time for observation has now elapsed, so that results at ambient room temperature of 25 ^{o}C can be presented for Epon Epoxy 828, Stycast Epoxy 3050, Thiokol Solithane and Sylguard 185.

The curve of pressure versus time for the cylindrical Epon 828 unit #3 is presented in Figure 1. Near the left-hand side is seen the steepest part of the curve plotted during the summer of 1973. From this the results in the earlier report[(1)] were computed. These results were premature and are now seen to be too large due to the fact that during the short few weeks of observation the Epon 828 was still outgasing internally into the evacuated cavity. The last pressure measurement in 1973 was taken on August 10, 1973. The unit was then not measured again until May 10, 1974. During this entire period of 9 months the pressure only increased by 10 torr. Then, when the breakdown voltage was measured more often again, the pressure increased more rapidly again; later, when the sparkgap was fired only once a month the increase was slower, namely 10 torr in

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(1) Measurements pertaining to Electrical Breakdown in Vacuum; Permeation of Air through Space Grade Insulations by Renate S. Bever; GSFC, Greenbelt, Maryland, X-761-73-353, August 1973

4 months. Averaging these results with those of a cubic Epon 828 unit one obtains for the permeation constant for Epon 828 with the aid of the equation developed in Reference (1).

$$\rho = 4 \times 10^{-11} \frac{\mathrm{STcm}^3 \cdot \mathrm{cm}}{\mathrm{sec} \cdot \mathrm{cm}^2\ \mathrm{cmHg}}$$

to one-significant figure accuracy.

Other methods were also tried. For instance, a Veeco thermocouple gage tube 4M was sealed into the top of a cylindrical unit of Epon 828 so that pressure could be read directly. The data confirmed the readings taken with the sparkgap but could not be carried far enough since above 20 torr the thermo-couple tube was not sensitive. Another method was to seal atmospheric pressure into the cavity and keep the unit in a vacuum system at 3×10^{-6} torr with 3 kv continuously on the sparkgap. No breakdown occured over a period of over 2 months, and when the unit was removed from the vacuum and tested to see where it broke down, it was found that no appreciable drop in gas pressure had occured inside the unit in the 2 months. All of this verifies the fact that the permeation of air through Epon 828 epoxy is indeed very slow.

It is even slower for the Stycast 3050 as seen in Figure 2. Again a thermo-couple tube used as a pressure measuring device instead of the sparkgap verified results with the sparkgap, and so did the method of sealing atmospheric pressure into the cavity and placing it into a vacuum system for 2 months. The results computed for Stycast 3050 give

$$P = 7\times10^{-12} \frac{\text{Stcm}^3 \cdot \text{cm}}{\text{sec cm}^2 \cdot \text{cmHg}}$$

Figure 3 shows the curve for Solithane. There

$$P = 5 \times 10^{-10} \frac{\text{Stcm}^3 \cdot \text{cm}}{\text{sec cm}^2 \cdot \text{cmHg}}$$

Figure 4 is a composite curve for Sylguard 185. The composite is made up of data points taken on two units and after several successive evacuations of the same unit

$$P \text{ for Sylguard 185} = 60 \times 10^{-9} \frac{\text{Stcm}^3 \cdot \text{cm}}{\text{cm}^2 \text{ sec cmHg}}$$

as computed from the low pressure end of the curve. Since the Sylguard permeation took only a few days, the reverse permeation was attempted with this material. Sealing one atmosphere into the cavity and placing it in vacuum caused the pressure to drop from 760 torr to 60 torr in 3 days and 5 hours for a Sylguard 185 unit, and from 760 torr to 93 torr for a Sylguard 184 unit.

During literature search for the adhesive study a table of gas permeability values of plastics and rubbers was discovered[2]. Although none of our space-grade insulation polymers appeared in this table, the permeation constants of related polymers in this table are of the same order of magnitude as the ones measured here.

The time for permeation depends on the ratio of volume of the cavity to area through which gas permeates, and it is therefore different for every different geometry of cavity or bubble trapped in a potting compound. Nevertheless, one can say in summary that for gas pressure in a trapped bubble to drop from atmospheric to the troublesome corona region would take years for Stycast 3050 and Epon 828, would take of the order of a month or so for Solithane and of the order of a few hours or a few days for a bubble in silicone rubber. It must be remembered, however, as is seen in Figures 1 through 4 that all 4 materials outgas internally very rapidly into any newly evacuated void that presents itself to them. Therefore, in all 4 materials, if a new void is created within them due to poor adhesion or due to cracking, then within these new voids, the pressure will rise from zero to a few torr of the corona region in tens of minutes or within an hour at least, and therefore good adhesion is of utmost importance to prevent this from happening.

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[2] Major, C. J. and Kammermeyer K, Modern Plastics, 39, # 11, 1962, p. 135.

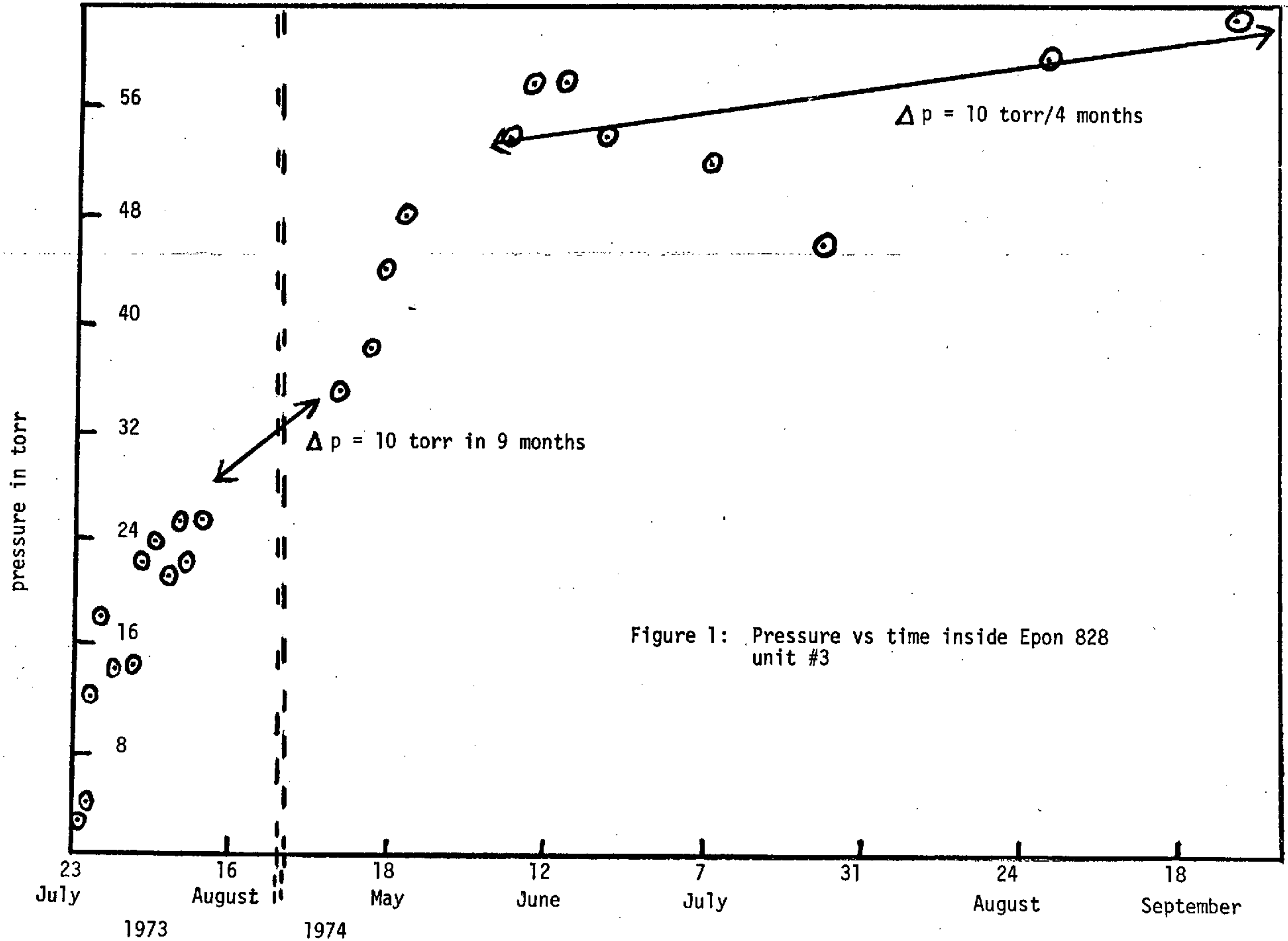

Figure 1: Pressure vs time inside Epon 828 unit #3

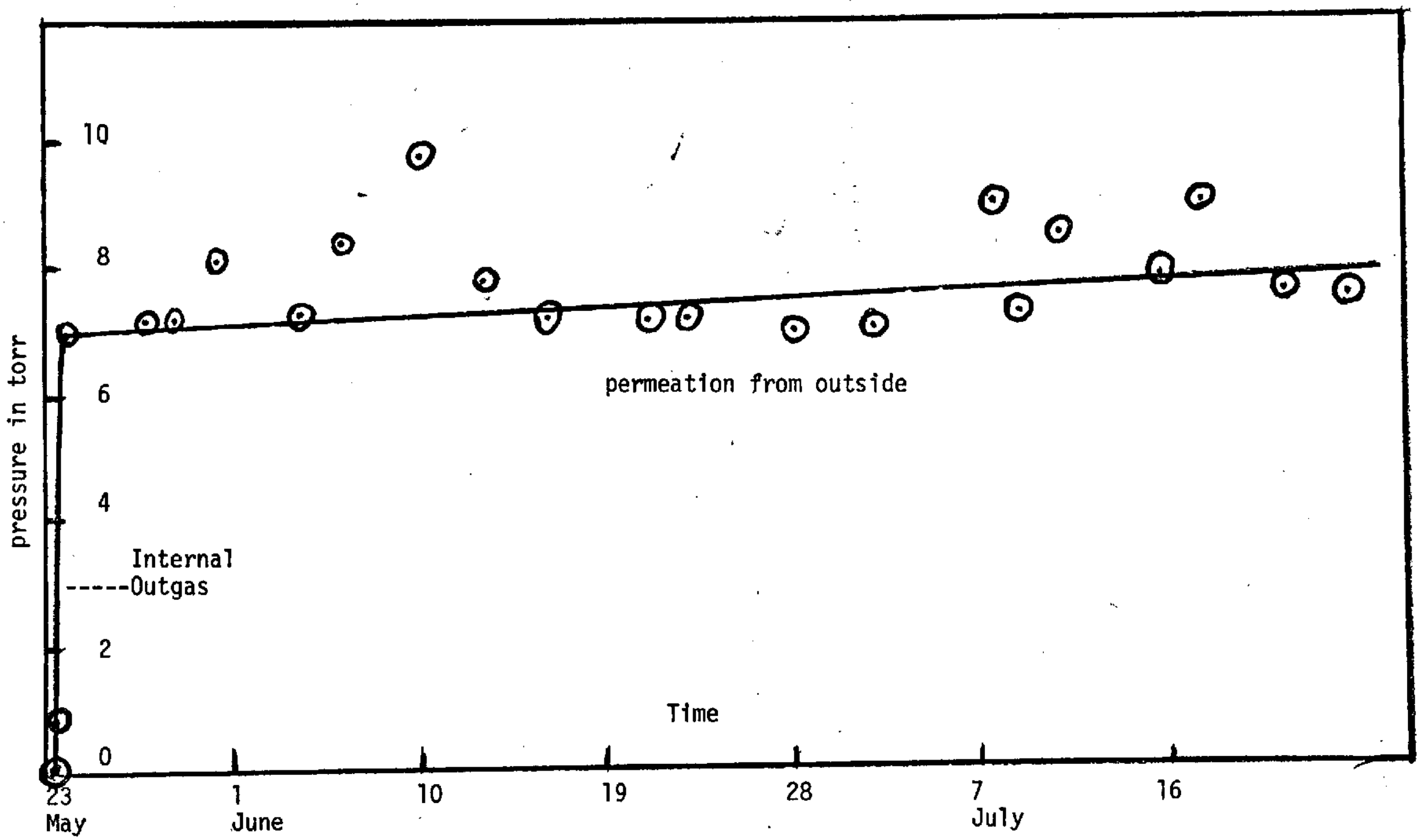

Figure 2: Pressure versus time for Stycast 3050

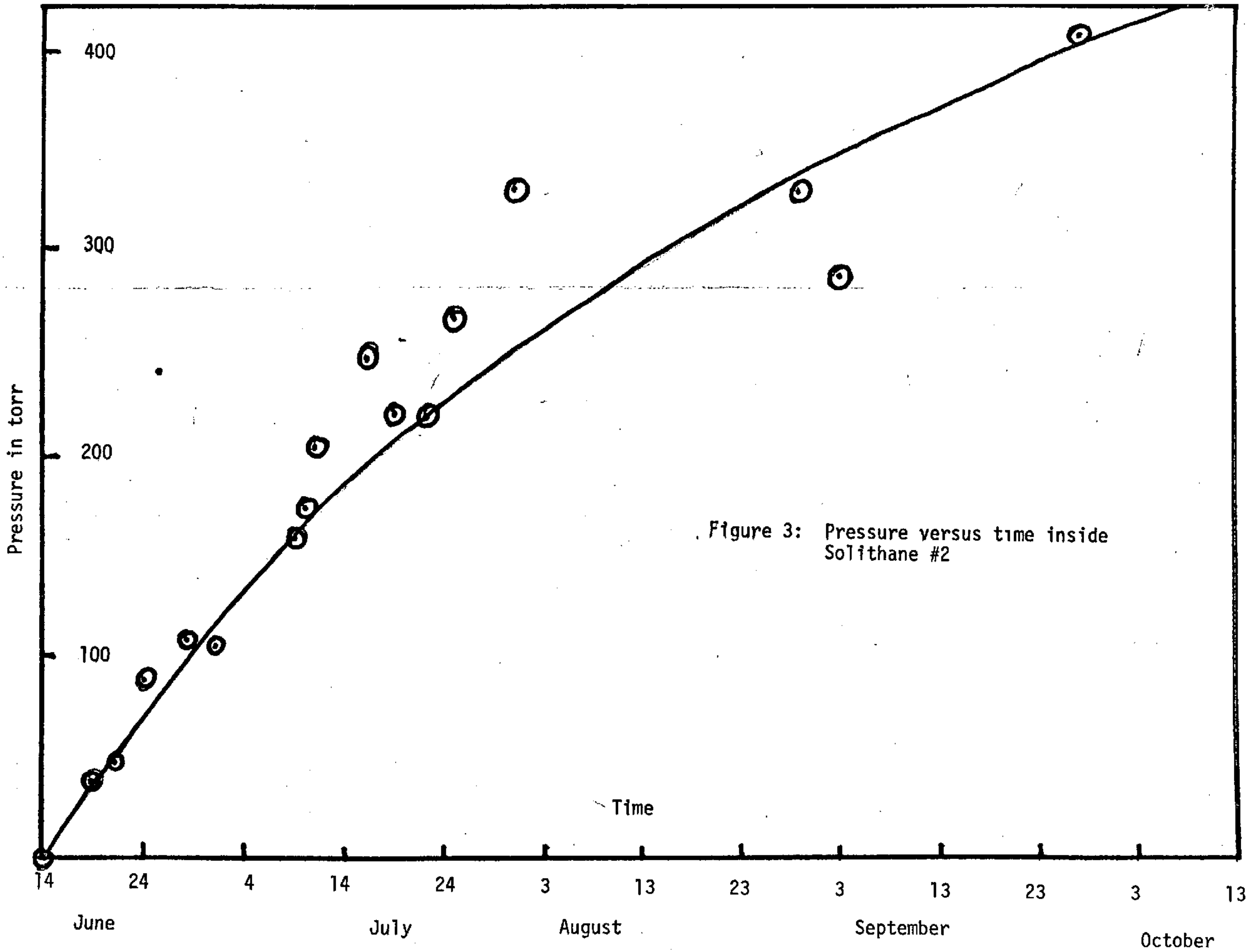

Figure 3: Pressure versus time inside Solithane #2

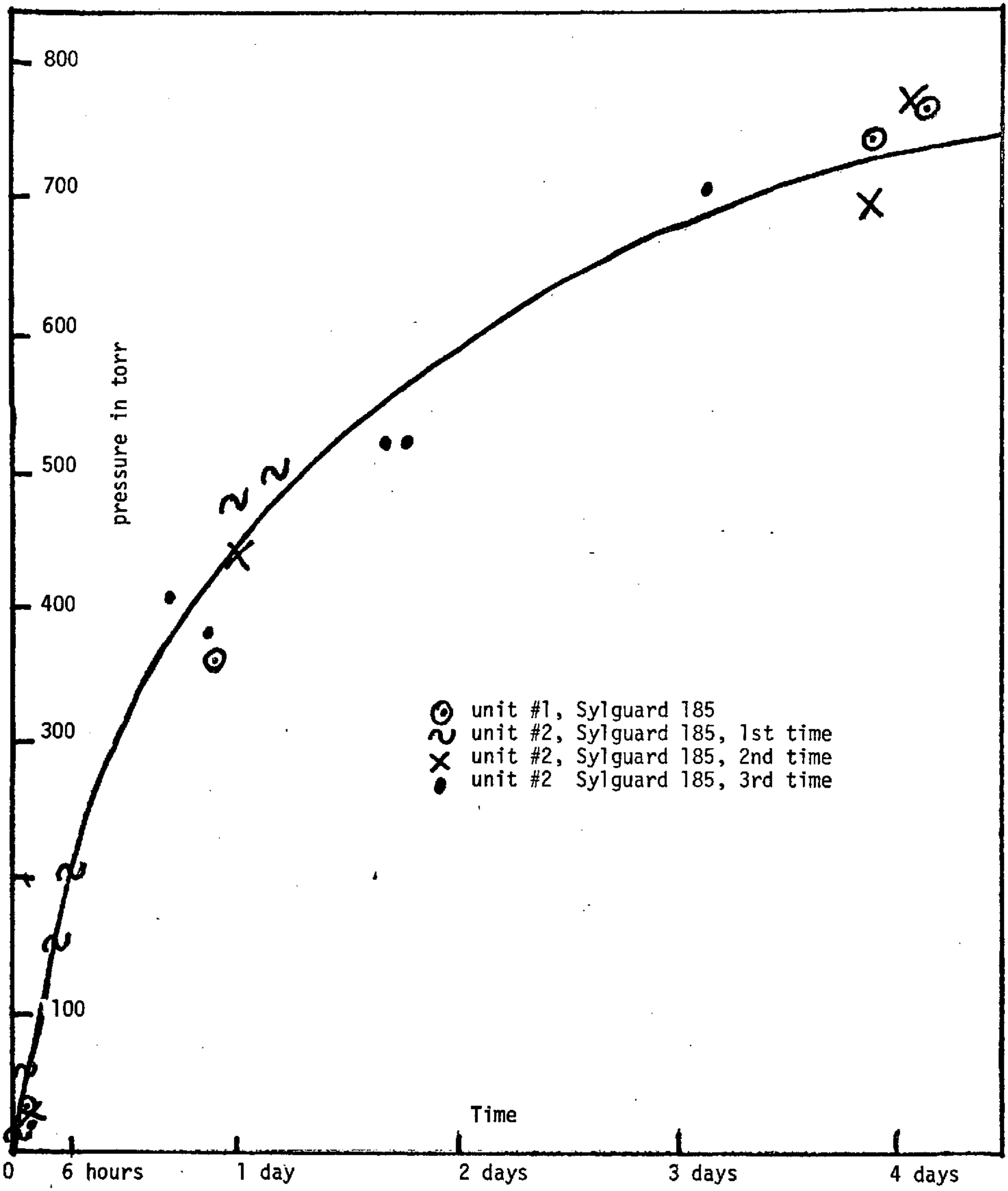

Figure 4: Pressure versus time for Sylguard 185

II Adhesive Study

This study is far from complete, and it will be carried on during the rest of the grant period. Much credit is due Mr. Carl Johnson of the Materials Section at GSFC for pulling the lap-shear specimens apart on the Instron Tensile Testing Machine. Dr. Ben Seidenberg, also of GSFC Materials Section, gave much helpful advice and supplied most of the materials.

The results of the study so far are best presented in Tables I, II and III.

Several conslusions can be drawn. For the solder plated on copper the adhesive forces to Epon 828 can be more than doubled by proper surface preparation. Grit blasting with a sharp grit, followed by ultrasonic cleaning gives best results. On the other hand what is best here is <u>not</u> best for preparation of glass-epoxy board. Simple alcohol spray seems to be superior here because it leaves the epoxy surface relatively undisturbed, and epoxy sticks best to epoxy.

Many attempts have been made to measure Epon 828 adhesive to porcelain alumina ceramic with a glue line of 0.020":

(a) Putting the ceramic as a strip across two pieces of glass epoxy board-the ceramic broke.

(b) Putting the ceramic as a filler between the 1/2" overlap of two 1" wide pieces of Aluminum or two pieces of 1" wide glass epoxy board. The sample then looks like a sandwich with the ceramic as the middle layer. The detachment always occured at the aluminum or glass epoxy surface at values much lower than measured for these materials. This seems at first strange until it is remembered that when one pulls lengthwise on the lap shear coupons, the thickness of the sandwich serves as a lever arm. Therefore, the thicker the glue line or the thicker the sandwich (by introducing a porcelain layer between the materials on which one pulls) the more the peel torque becomes effective. One therefore measures a mix of peel and lap shear and does not get true lap shear values of adhesion.

The highest values of detachment so far obtained were C7-750 psi, C8-640 psi, C9-680 psi. Therefore, we can state that the adhesive force of Epon 828 to porcelain ceramic is at least greater than twice the averages of these, namely 1360 psi.

It was also attempted to measure Epon 828 adhesion to ferrite, glue line 0.020". Again, the yokes of 4 ferrite horseshoe magnets were glued as straps across two 1/4" wide pieces of glass epoxy board with the Epon 828, 1/2" overlap. The ferrite cross-section actually broke in half at maximum loads of

142, 120, 147, 160 lbs, so that one can state that the Epon 828 adhesion to ferrite is certainly greater than 142 ÷ 1/8 psi or 1140 psi.

We are just beginning the measurements of adhesion of Solithane to various adherends. The adhesive strength to solder after alcohol spray or ultrasonic clean is of the order of only 100 psi, an order of magnitude weaker than Epon 828.

TABLE I

Adherend: 60% tin 40% lead solder, electroplated on Beryllium copper

Adhesive: Shell Epon 828/Miller Stephemson V-40

Specimen Number	Adhesive Thickness	Max Load	Shear Strength	Average	Standard Deviation	St.D./$\sqrt{n}$
	inches	lb	psi	psi		
Alcohol sprayed						
1-1	.017	515	1030			
1-3	.017	415	830			
1-4	.017	390	780			
1-5	.018	470	940	900	80	32
1-6	.017	455	910			
Alcohol sprayed, then sanded with SiC 320 paper, then alcohol sprayed						
2-1	.018	588	1180			
2-2	.019	570	1140	1200	50	20
2-3	.018	590	1180			
2-4	.016	642	1280			
2-5	.019	568	1140			
2-6	.018	630	1260			
Ultrasonic clean with Freon TF						
3-1	.020	368	740			
3-2	.020	400	800			
3-4	.021	590	1180	1000	230	92
3-5	.019	655	1310			
3-6	.021	490	980			

(Continuation of Table I)

Adherend: 60% Tin - 40% lead solder, electroplated on Beryllium copper

Adhesive: Shell Epon 828/Miller Stephemson V-40

Specimen Number	Adhesive Thickness	Max Load	Shear Strength	Average	Standard Deviation	St.D./$\sqrt{n}$
	inches	lb.	psi	psi		
Ultrasonic clean Freon TF, grit blasted with glass-balls, ultras. clean						
4-1	.020	862	1720			
4-2	.022	710	1420			
4-3	.017	561	1120			
4-4	.020	580	1160	1360	210	84
4-5	.022	750	1500			
4-6	.020	620	1240			
Vapor degreased with trichloroethane						
5-1	.021	460	920			
5-2	.018	812	1620			
5-3	.019	585	1170			
5-4	.023	672	1340	1170	260	104
5-5	.019	552	1100			
Ultrasonic clean, wipe with paper towel, ultrasonic clean Freon TF						
6-1	.018	605	1210			
6-2	.018	862	1720			
6-3	.020	526	1050			
6-4	.020	525	1050	1180	250	100
6-5	.020	520	1040			
6-6	.020	502	1000			
glue line half as thick						
6-7	.008	680	1360	1460	100	40
6-8	.011	778	1560			

(Continuation of Table I)

Specimen Number	Adhesive Thickness	Max Load	Shear Strength	Average	Standard Deviation	St.D./$\sqrt{n}$
	inches	lb.	psi	psi		
Vapor degreased with trichloroethylene						
7-1	.019	575	1150			
7-2	.019	365	730			
7-3				960	200	80
7-4	.019	585	1170	Low value not under-		
7-5	.019	390	780	stood, should be repeated		
Vapor Degrease with trichloroethylene, blast with glass balls, then vapor degrease again						
8-1	.019	1070	2140			
8-2	.019	760	1520			
8-3	.019	950	1900			
8-4	.019	830	1660	1810	230	92
8-5	.019	960	1900			
8-6	.019	830	1660			
Ultrasonic clean Freon TF, bombard with sharp Black Beauty grit 80 mesh, ultras. clean again						
glue line half as thick						
9-1	.010	1050	2100			
9-2	.010	1020	2040	2100	50	29
9-3	.010	1090	2180			
9-4	.020	870	1740			
9-5	.020	1040	2080	1950	120	70
9-6	.020	960	1920			
As received, 1/4" wide strips						
0-1	.020	105x4	840			
0-2	.020	96x4	768	700	105	52
0-3	.020	89x4	712			
0-4	.020	62x4	496			

TABLE II

Adherend: GLASS-EPOXY BOARD

Adhesive: EPON 828/MILLERSTEPHENSON V-40

Specimen Number	t	Max Load	Shear Strength	Average	Standard Deviation	St.D./$\sqrt{n}$
	inches	lb.	psi	psi		
Ultrasonic clean, Freon TF						
glue line half as thick						
E-1-1	.008	1220	2440			
E-1-2	.009	1220	2440			
E-1-3	.007	980	1960	2450	300	120
E-1-4	.008	1140	2280			
E-1-5	.009	1280	2660			
E-1-6	.008	1460	2920			
Ultrasonic clean, Freon TF, sandblasted with glass balls, ultrasonic clean						
E-2-1	.019	710	1420			
E-2-2	.019	620	1240			
E-2-3	.019	800	1600	1430	240	96
E-2-4	.019	600	1200			
E-2-5	.019	940	1880			
E-2-6	.019	620	1240			
Vapor degrease, trichloroethylene						
E-3-1	.019	760	1520			
E-3-2	.019	540	1080			
E-3-3	.019	840	1680	1350	240	96
E-3-4	.019	562	1120			
E-3-5	.019	790	1580			
E-3-6	.019	580	1160			
Vapor degrease, trichloroethylene, sandblast glass balls, vapor degrease						
E-4-1	.019	660	1320			
E-4-2	.019	740	1480			
E-4-3	.019	670	1340	1380	80	32
E-4-4	.019	630	1280			
E-4-5	.019	750	1500			
E-5-6	.019	690	1380			

(Continuation of Table II)

Specimen Number	t	Max Load	Shear Strength	Average	Standard Deviation	St.D.
	inches	lb.	psi	psi		
Alcohol Spray						
E-5-1	.019	870	1740			
E-5-2	.019	800	1600			
E-5-3	.019	1170	2340	2000	260	104
E-5-4	.019	1090	2180			
E-5-5	.019	940	1980			
E-5-6	.019	1110	2220			
Ultrasonic clean, Freon TF						
E-6-1	.019	980	1960			
E-6-2	.019	1000	2000			
E-6-7	.019	980	1960	1900	100	40
E-6-4	.019	920	1940			
E-6-5	.019	890	1780			
E-6-6	.019	870	1740			
Ultrasonic clean. Freon TF						
E-7-1	.030	640	1280			
E-7-2	.030	930	1860	1710	290	170
E-7-3	.030	1000	2000			

TABLE III

Adherend: 60-40 solder electroplated on berylium copper

Adhesive: Solithane

Thickness: 0.010 inches

Alcohol Spray

	Max. Load lb.	Shear Strength psi
S-A-1	50	100
S-A-2	58	116
S-A-3	55	110
S-A-4	52	104
S-A-5	60	120

Ultrasonic Clean Freon TF

	Max. Load lb.	Shear Strength psi
S-U-1	41	82
S-U-3	60	120
S-U-4	54	108
S-U-5	25	50
S-U-6	No results	

III BEHAVIOR OF THE SERIES 600 HIGH VOLTAGE CONNECTORS AT VARIOUS PRESSURES. MANUFACTURED BY REYONLDS INDUSTRIES, INC., 5005 McCONNELL AVE., LOS ANGELES, CALIF. 90066.

These high voltage connectors are internally completely clad by diallyl phtalate and are made "vacuum tight" at the cable junction by tiny O-rings. The experimental set-up for investigation largely having been designed and assembled by Dr. John F. Sutton of GSFC, code 325.1, is shown in Figure 5. The discriminator was set so that the scaler counted all counts above 3×10^{-14} coulombs and the window counter would register counts between 3×10^{-14} and 3×10^{-13} coulombs. The latter generally comprised about 80% of all counts. The high voltage applied was 3 kilovolts.

When the apparatus was first encountered, it was electrically noisy so as to obscure the behavior of the test connector in the vacuum chamber. Potting of the 10 Megohm resistor at A and the 0.001 mf condenser at O gave no improvement. However, the following steps did improve the performance:

(a) Replacing the never-outgased Reynolds connectors between points AB and BD by a solid piece of buss wire.

(b) Replacing the never-outgased Reynolds connectors between points BE and F with an RG-59-cable-high vacuum feed-through, continuous from points B to P, where it was spliced onto the Reynolds cable, the splice being solid-potted with Epon 828 epoxy.

(c) The vacuum chamber was then outgased at 10^{-3} torr for 2 days.

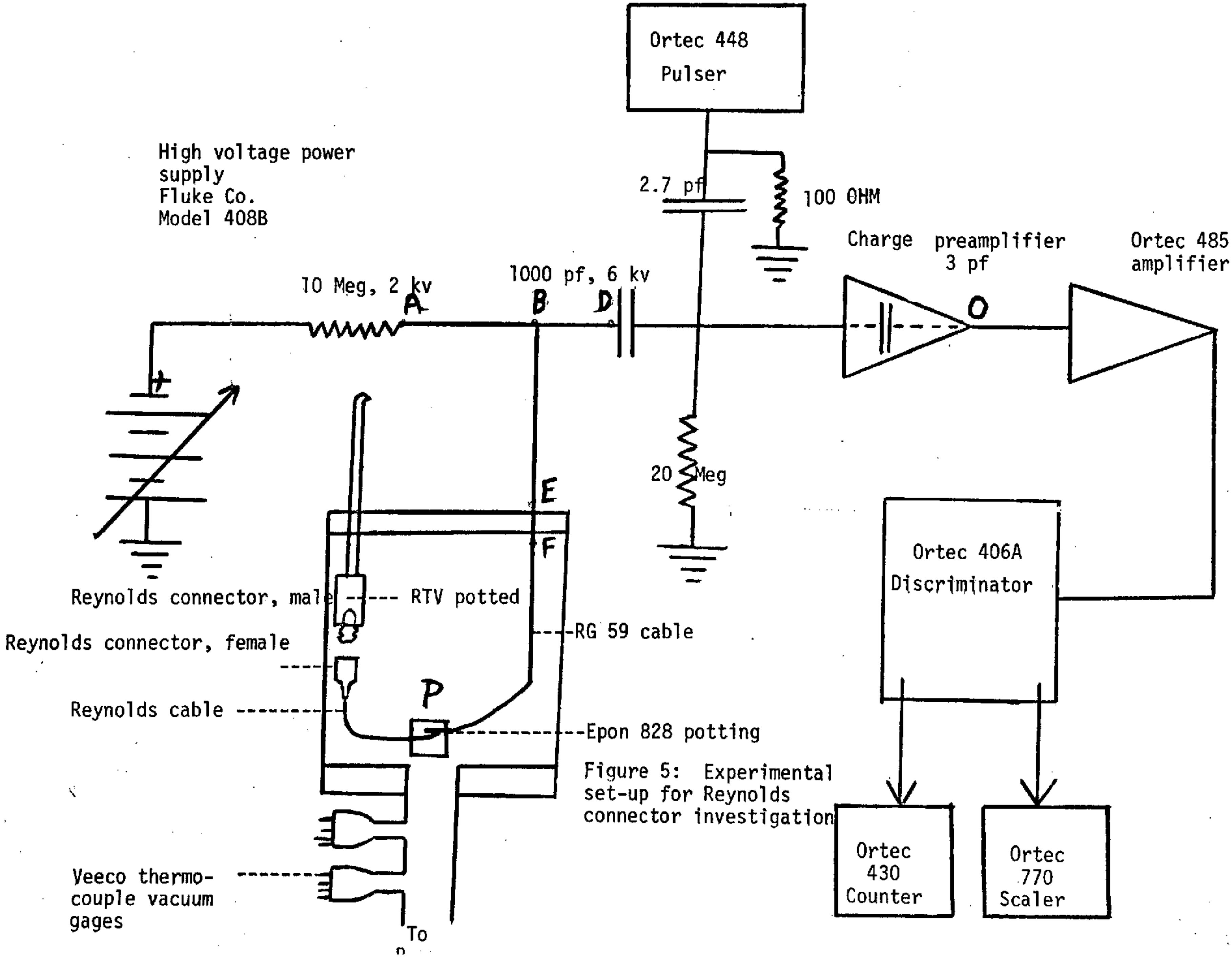

Figure 5: Experimental set-up for Reynolds connector investigation

The above steps resulted finally in zero count-rate at 10^{-3} torr and gave a starting point for the investigation.

Several dozen graphs of count-rate versus time at 3000 volts. with the Reynolds connector in the vacuum chamber filled at various pressures, are available. These have not yet been reproduced and inked for inclusion in this report. This will be done in a later report. However, the conclusions so far may be summarized as follows:

(1) After outgasing at 10^{-3} torr for 2 days the Reynolds connectors are noise free both below and above the corona region.

(2) In the corona region, which is between about 1.5 torr to 10 torr for these connectors, they indeed do suppress catastrophic breakdown, even after having been opened and closed innumerable times. This is amazing to behold.

(3) In the corona region, the connectors do show some noise counts, in general about 20 counts per 5 minute interval.

(4) This can be cut in half to 10 counts per 5 minute interval by potting the cable ends with Stycast 3050 or Solithane, where the ends interface with the connector proper.

(5) The noise ~~counts appear~~ in two types of modes: Either a steady "dribble" of single counts or a period of complete quiet for several minutes followed by a multi-count burst. The latter behavior occured

most often after very long outgasings.

(6) Looking at the pulses at the output of the operational amplifier at O with a Tektronix "memory" oscilloscope it became evident that the multiple bursts are usually very energetic single counts that set up high voltage shock oscillations in the circuit.

The following further steps are now being undertaken:

(a) See if a filter network will eliminate or integrate the multiple bursts.

(b) Replace the present Reynolds connector with a new one, and then do long-term corona testing. That is, trap corona region pressure in the connector and apply 2500 volts continuously for 3 to 4 weeks to see if the noise count stays constant or increases.

(c) Design a long-term test to see at what time rate gas leakage occurs out of the connector when a differential of one atmosphere of pressure exists between the inside and outside of the connector.

This experimental apparatus can of course be used to check on the electric noise-level of circuit components. By disconnecting the test chamber at B and by inserting different capacitors in place of the 0.001 mf one at point D one can check the relative noisiness of capacitors. For instance, one can verify this way that plastic, liquid filled or so-called Glassmike condensers are more quiet than ceramic ones.

By connecting 1 ft lengths of different types of cables at point B, cut ends potted, one can find which type of cable is the most quiet, the winner here being solid center-wire type such as RG 59 cable.

IV. The strain gage instrumentation has arrived. Solid potting of a small circuit, followed by temperature cycling and accompanying strain gage measurements is now being instituted.

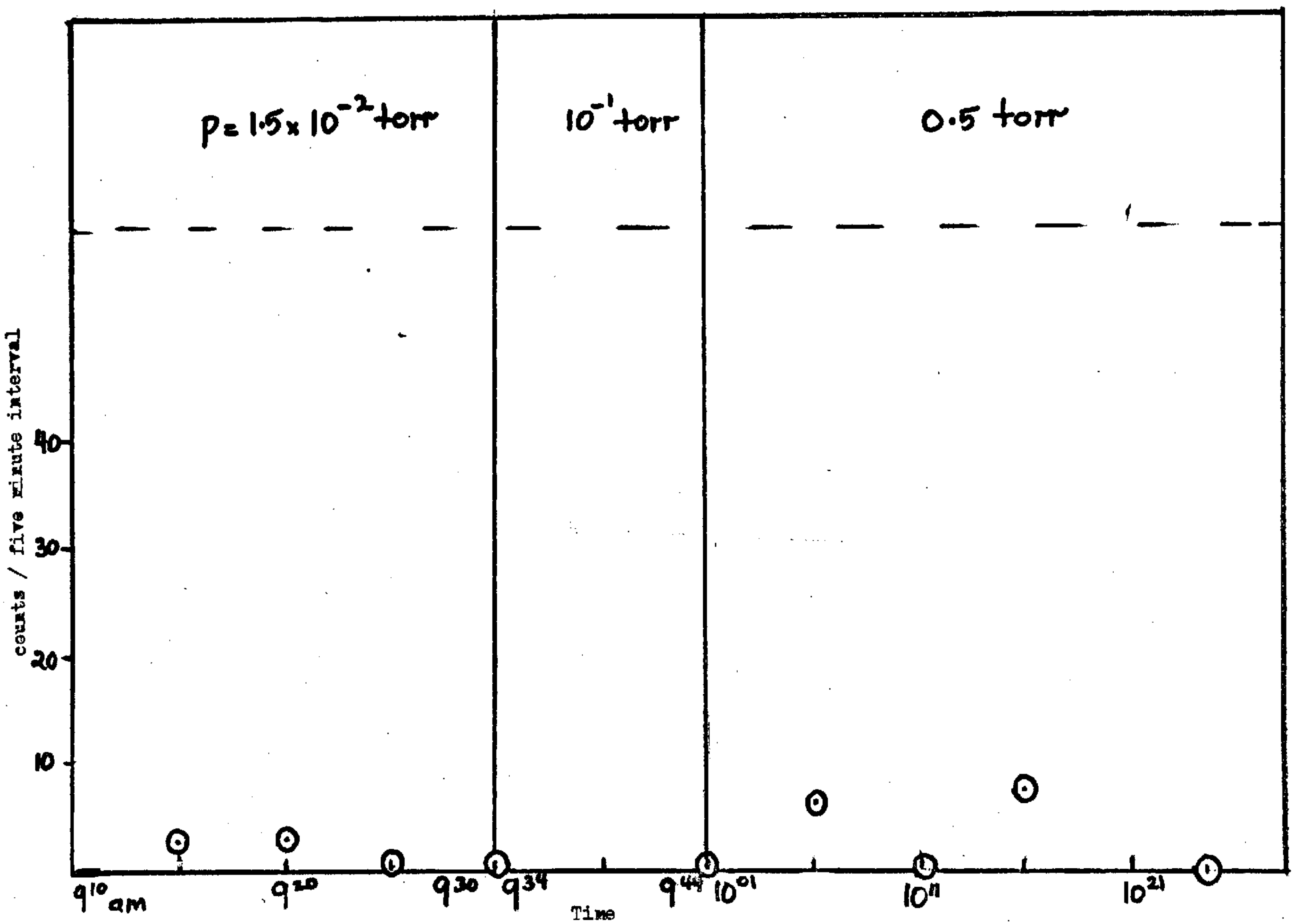

Figure 6a: August 15,74. Series 600 Reynolds connector, filled at different pressures with ordinary air.

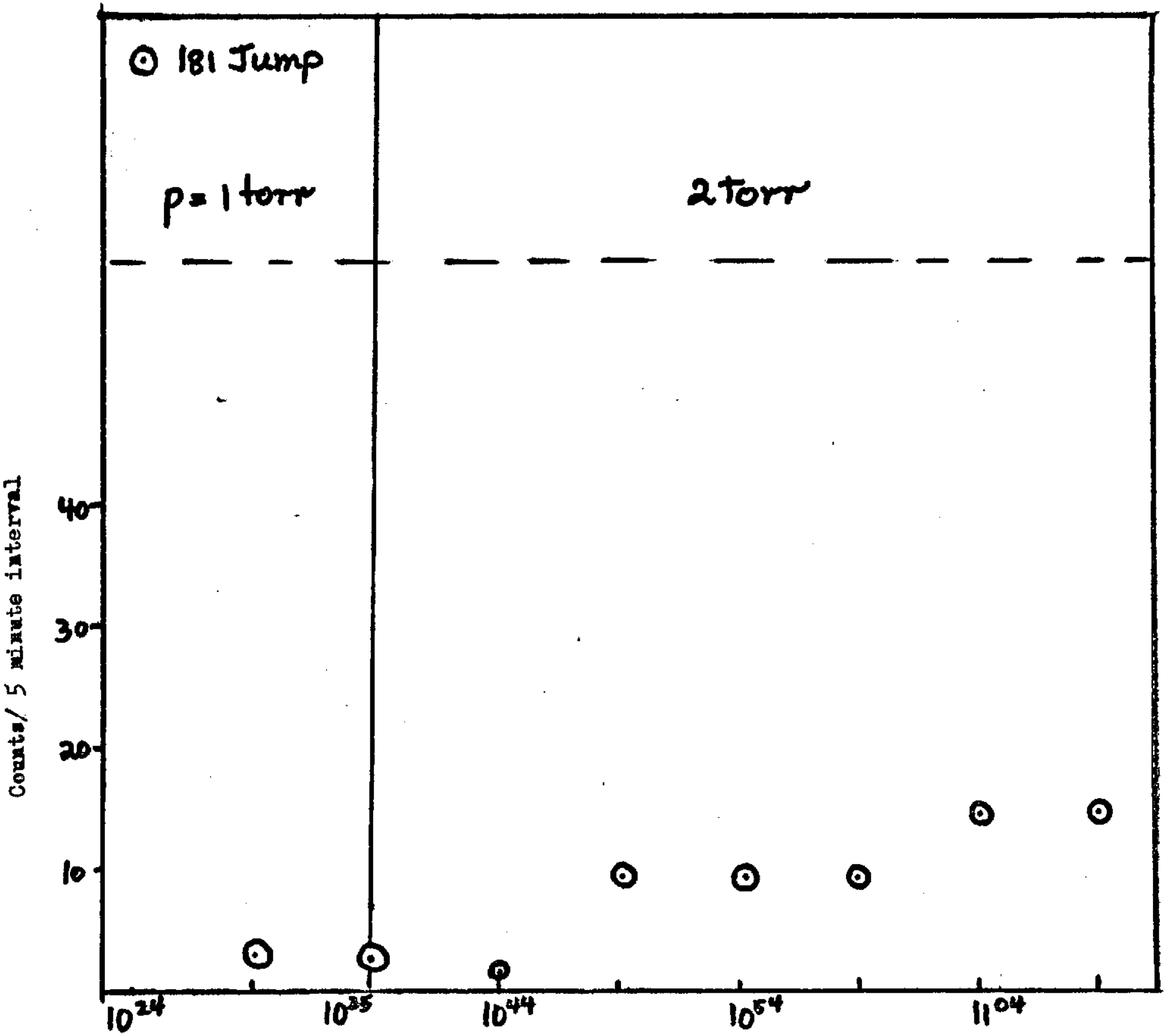

Figure 6b: August 15,74: Series 600 Reynolds connector, filled at different pressures with ordinary air

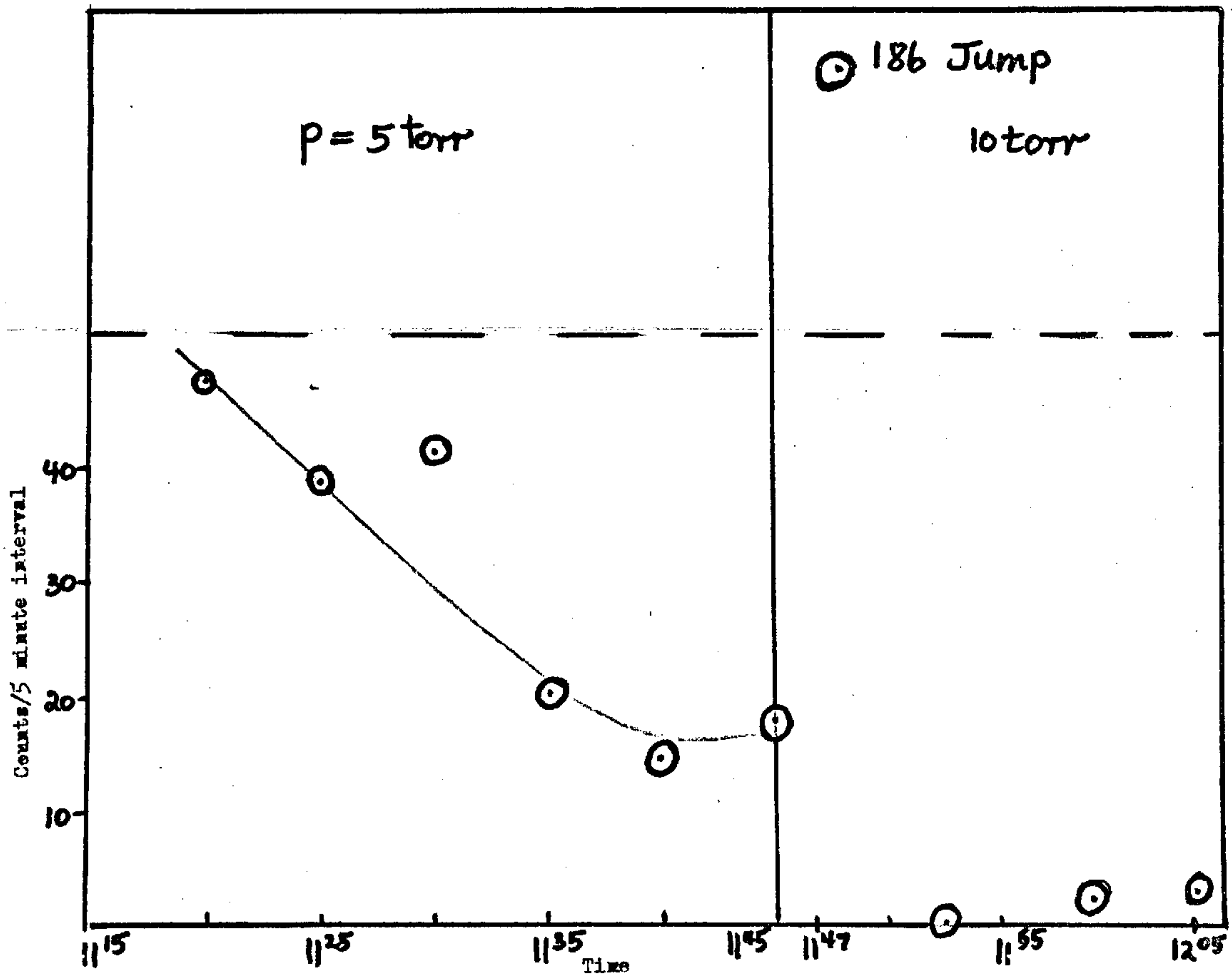

Figure 6e: August 15, 74: Series 600 Reynolds connector, filled at different pressures with ordinary air

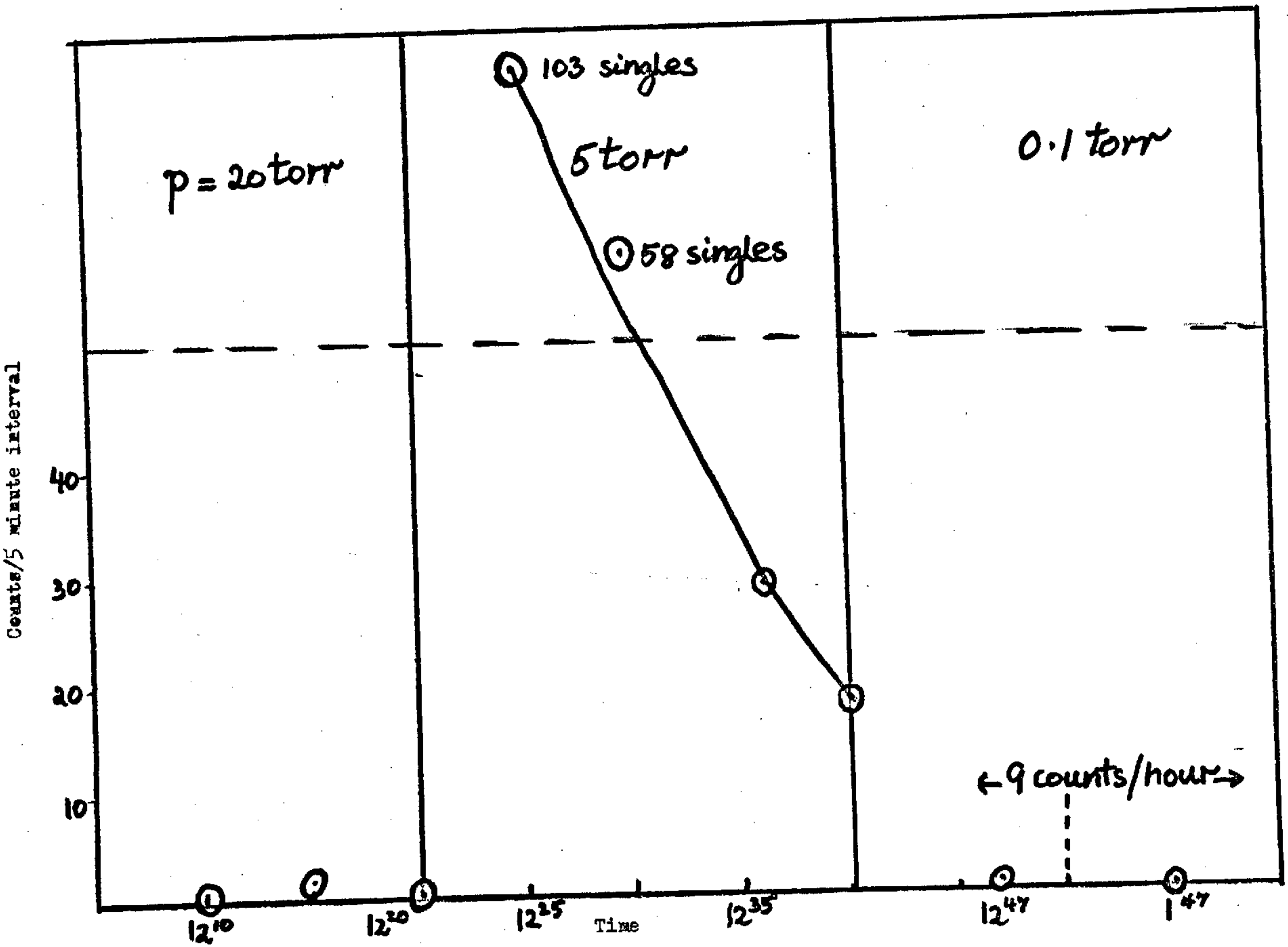

Figure 6d: August 15,74: Series 600 Reynolds connector; filled at different pressures with ordinaty air.

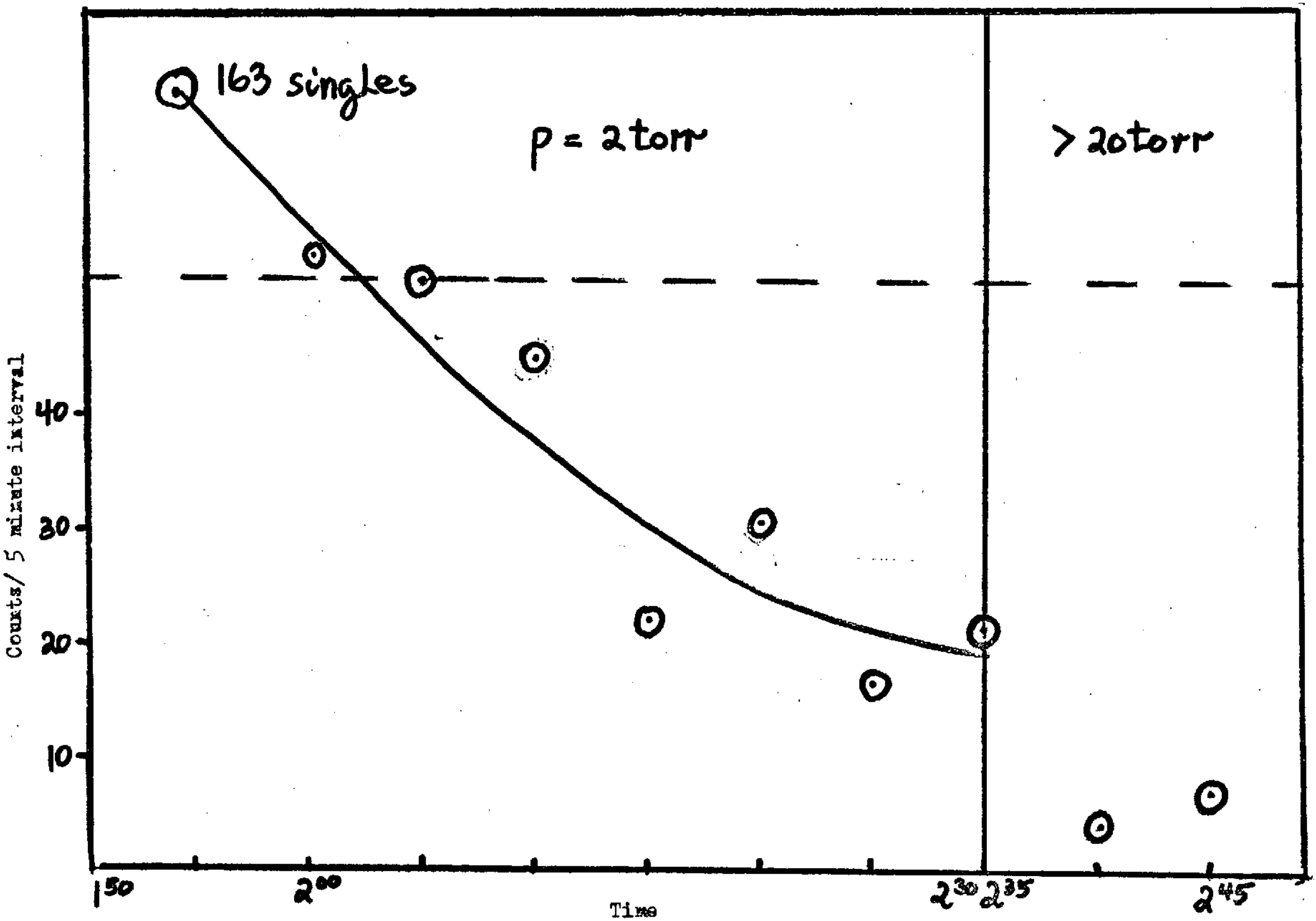

Figure 6e: Series 600 Reynolds connector, filled at different pressures with ordinary air, August 15, 74

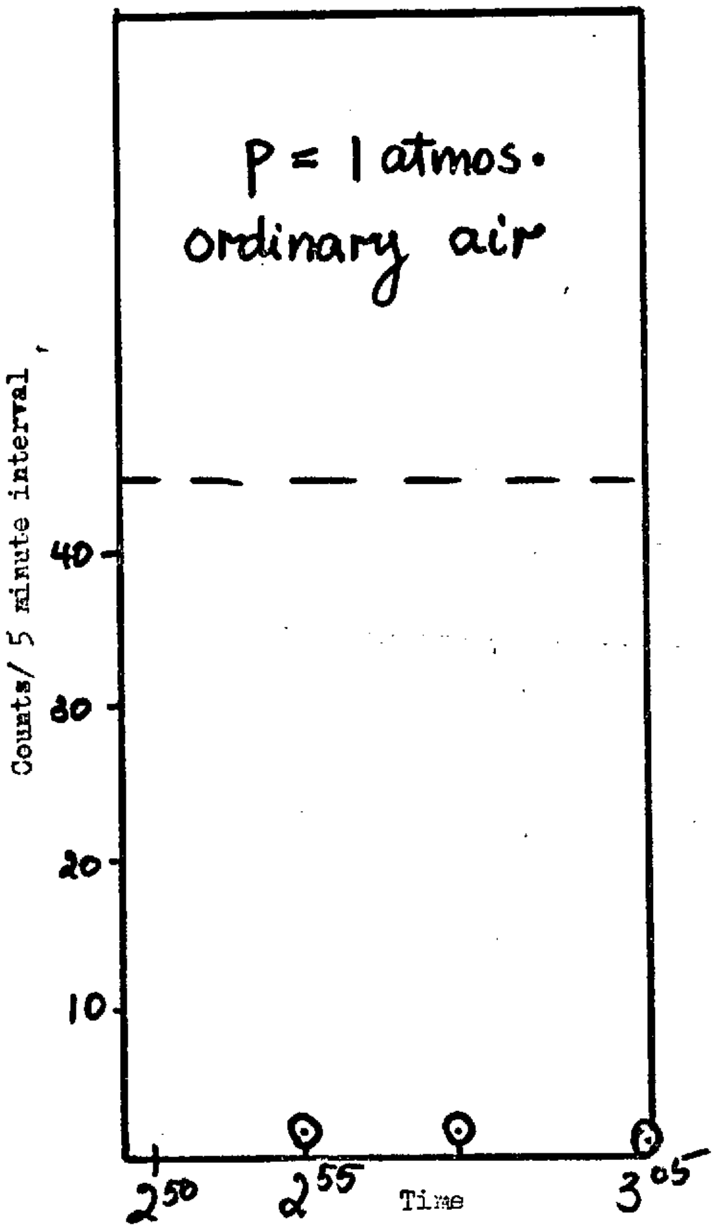

Figure 6f: August 15, 74: Series 600 Reynolds connector, filled at different pressures with ordinary air

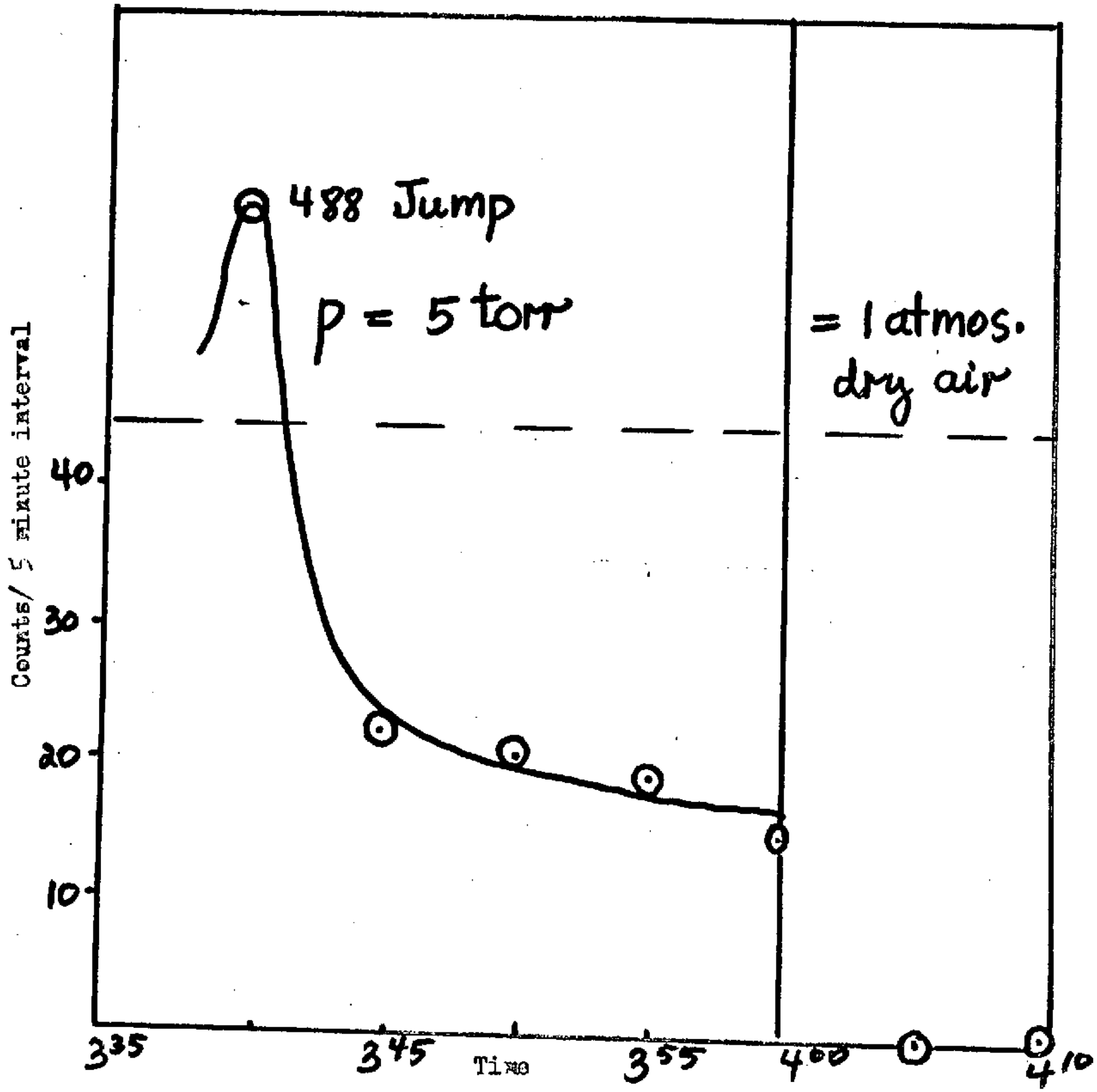

Figure Ia: Series 600 Reynolds connector, August 15,74, filled at different pressures with dessicant dried air

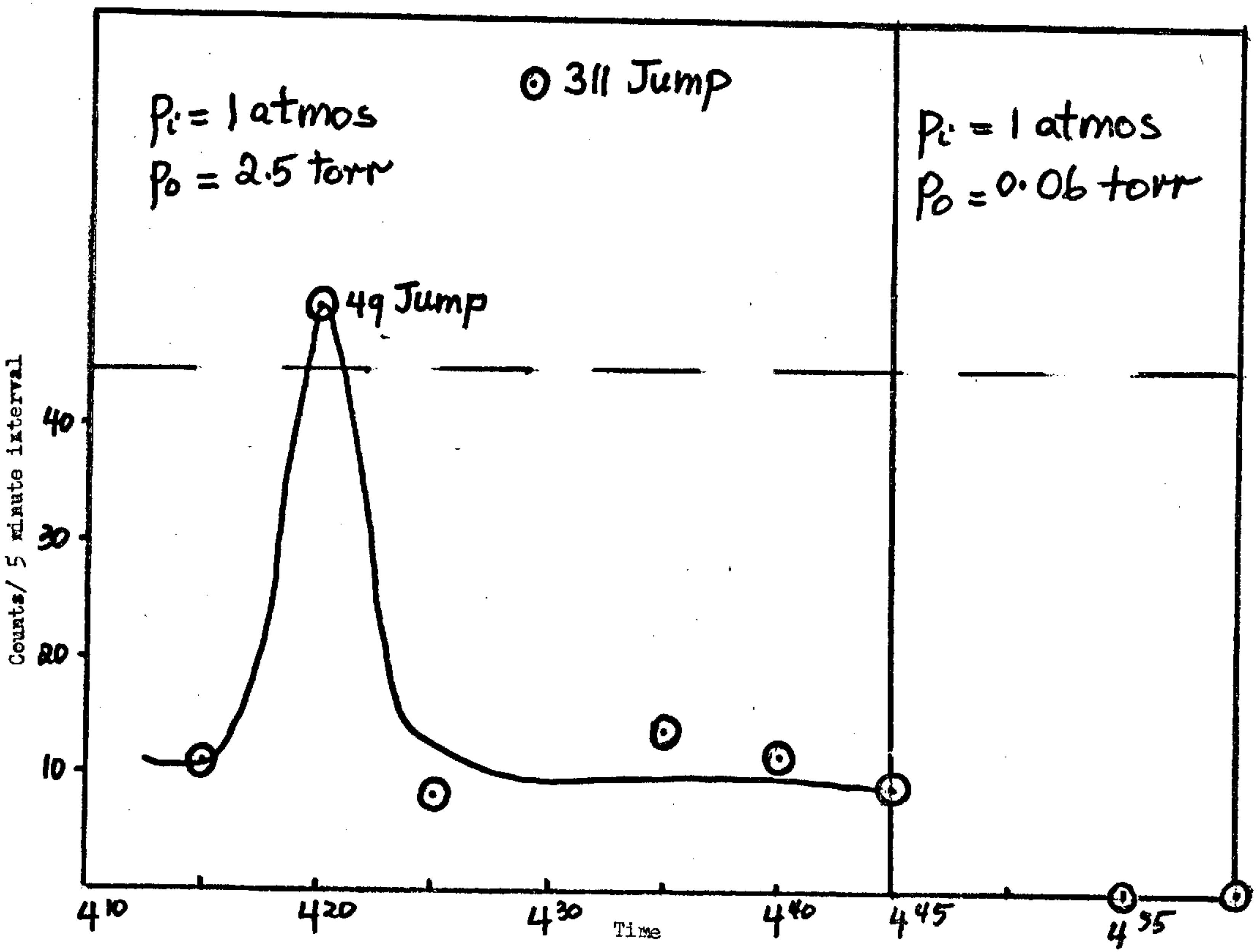

Figure 7b: August 15, 74: Series 600 Reynolds connector, filled at different pressures with dessicant dried air

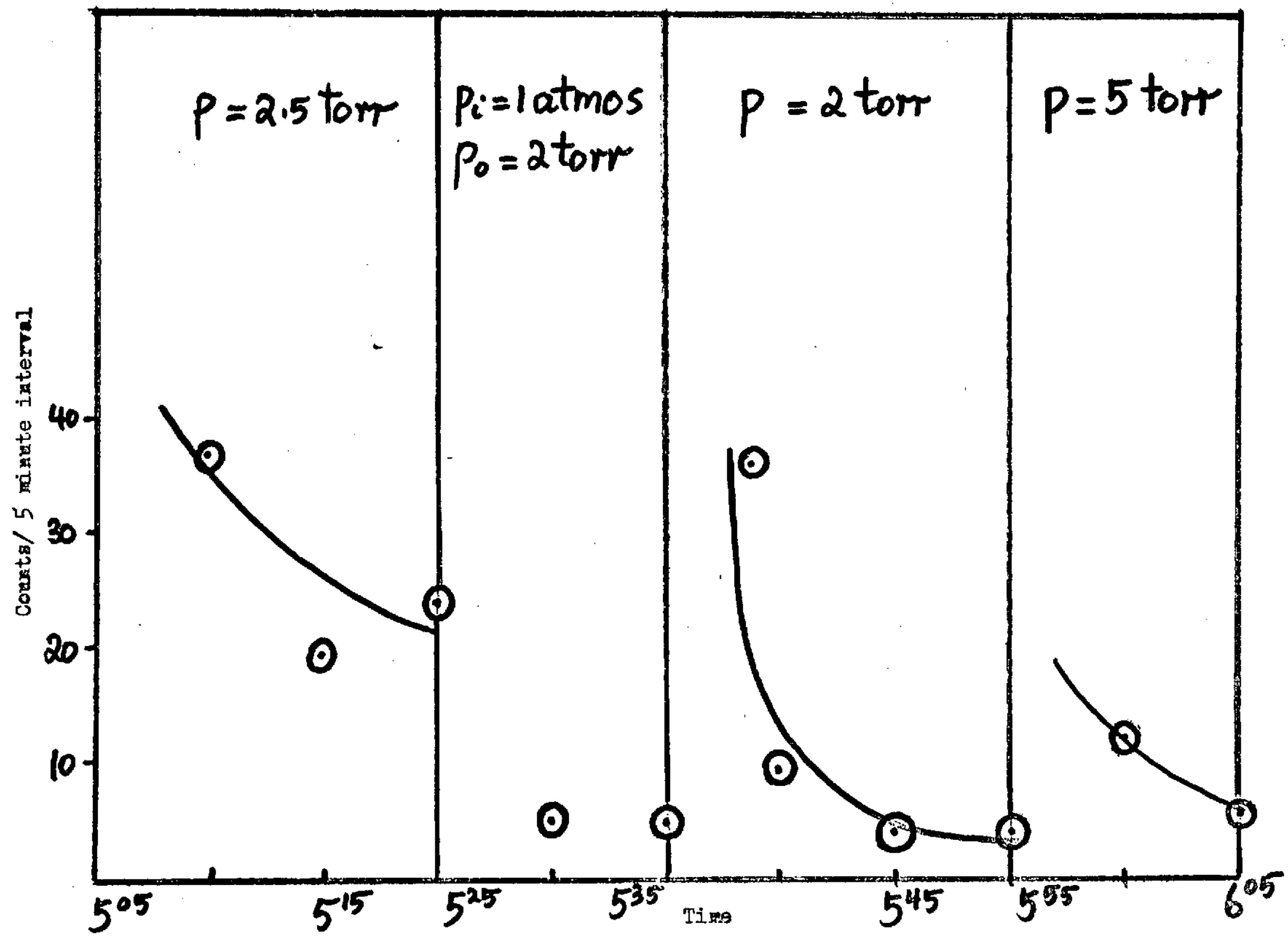

Figure 7a: August 15,74: Series 600 Reynolds connector, filled at different pressures with dessicant dried air

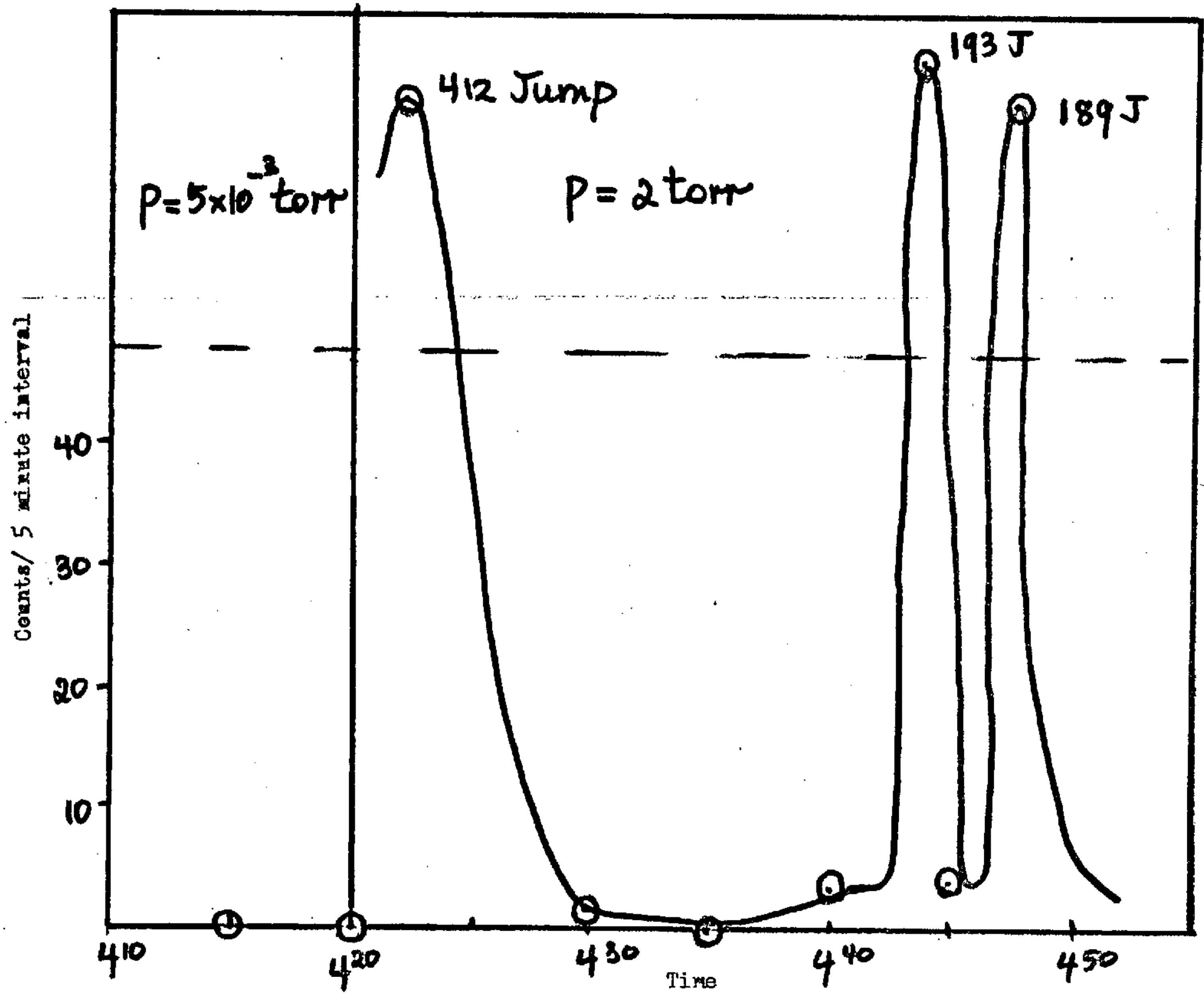

Figure 8a: August 26,74: Series 600 Reynolds connector after 5 days of continuous outgasing, filled at different pressures with dessicant dried air

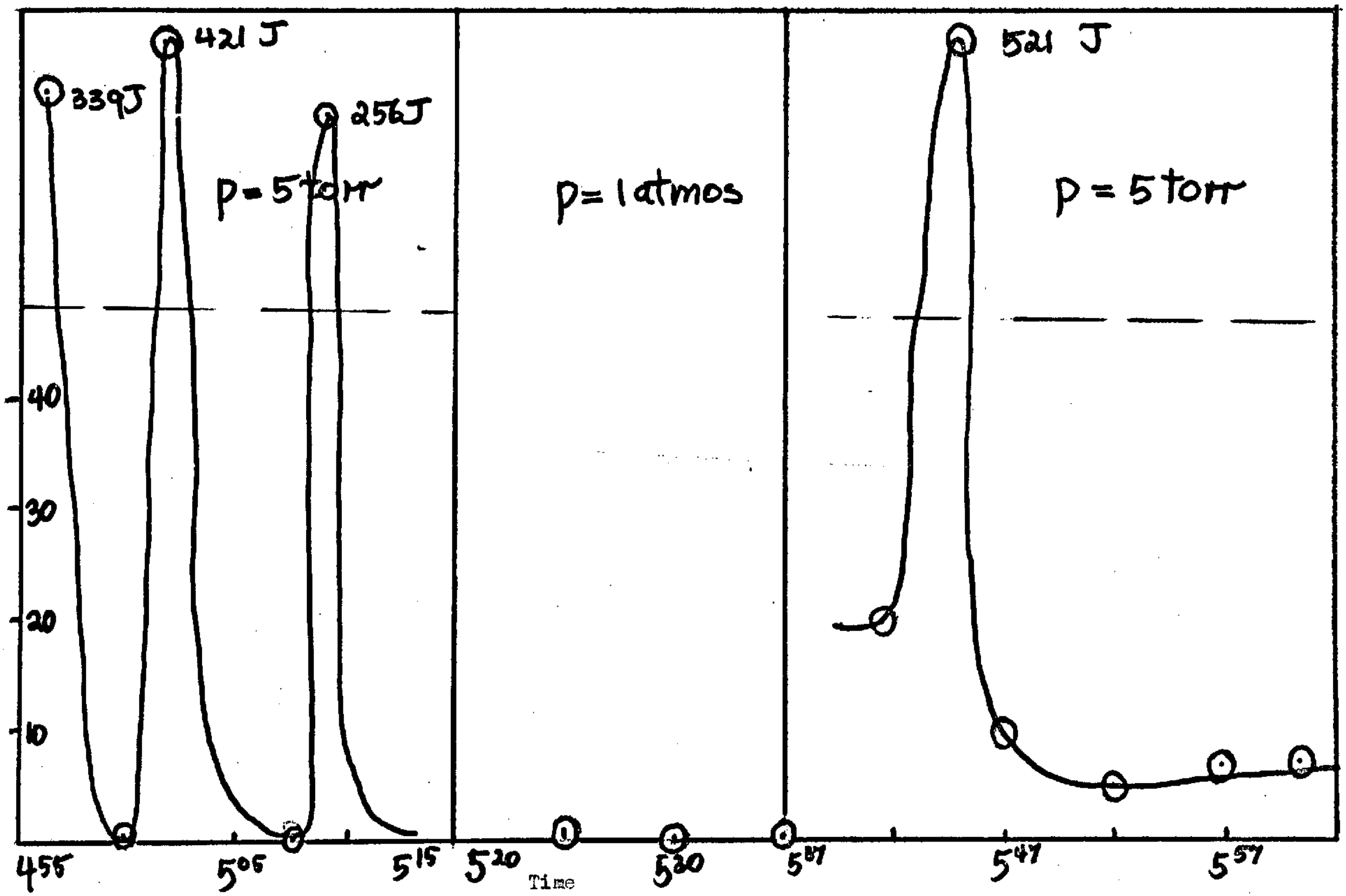

Figure 8b: August 26,74: Series 600 Reynolds connector after 5 days of continuous outgasing, filled at different pressures with dessicant dried air.

Aug 27

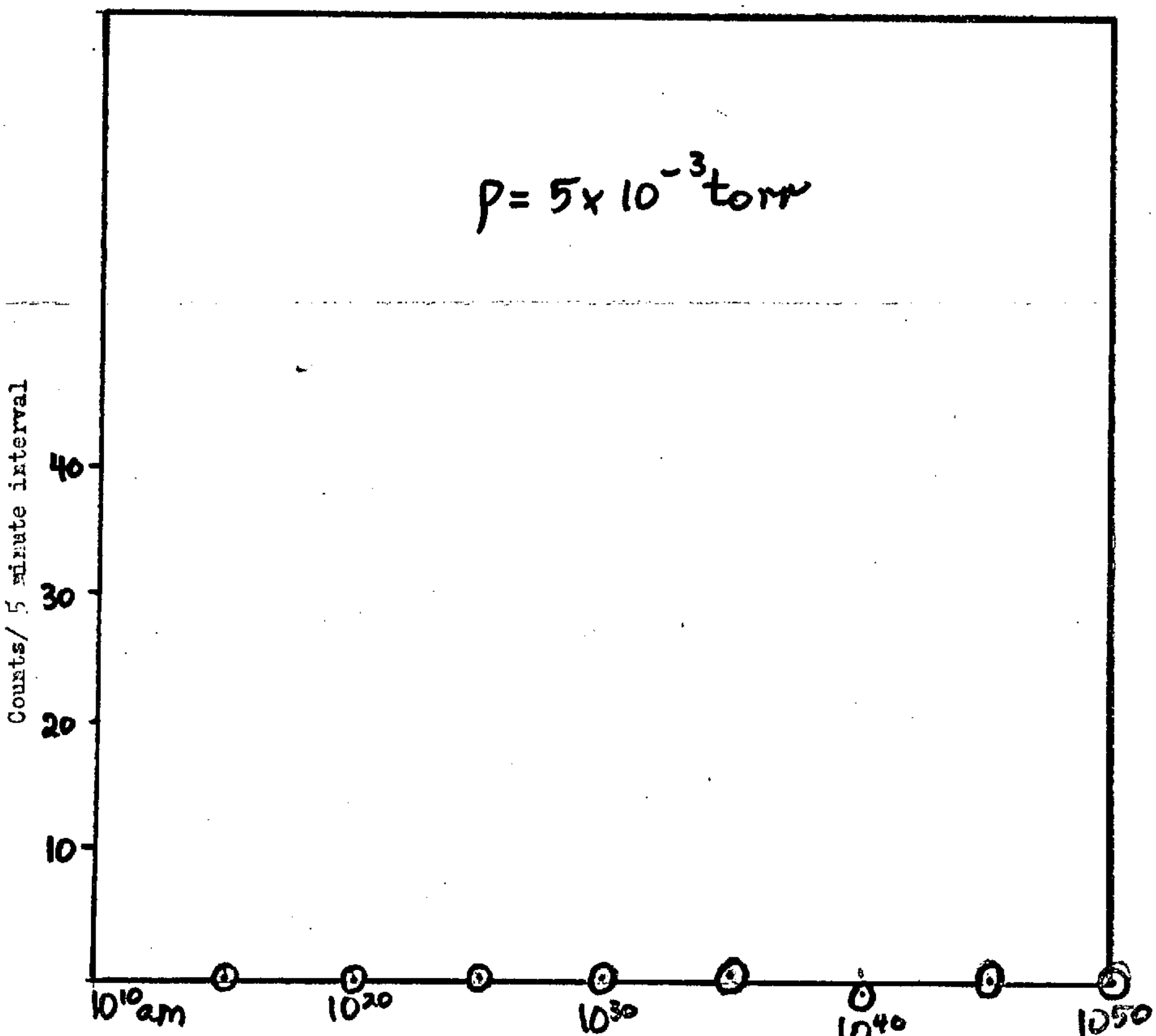

Figure 9a: August 27,74, Series 600 Reynolds connector after 5 days of continuous outgasing, filled at different pressures with dessicant-dried air

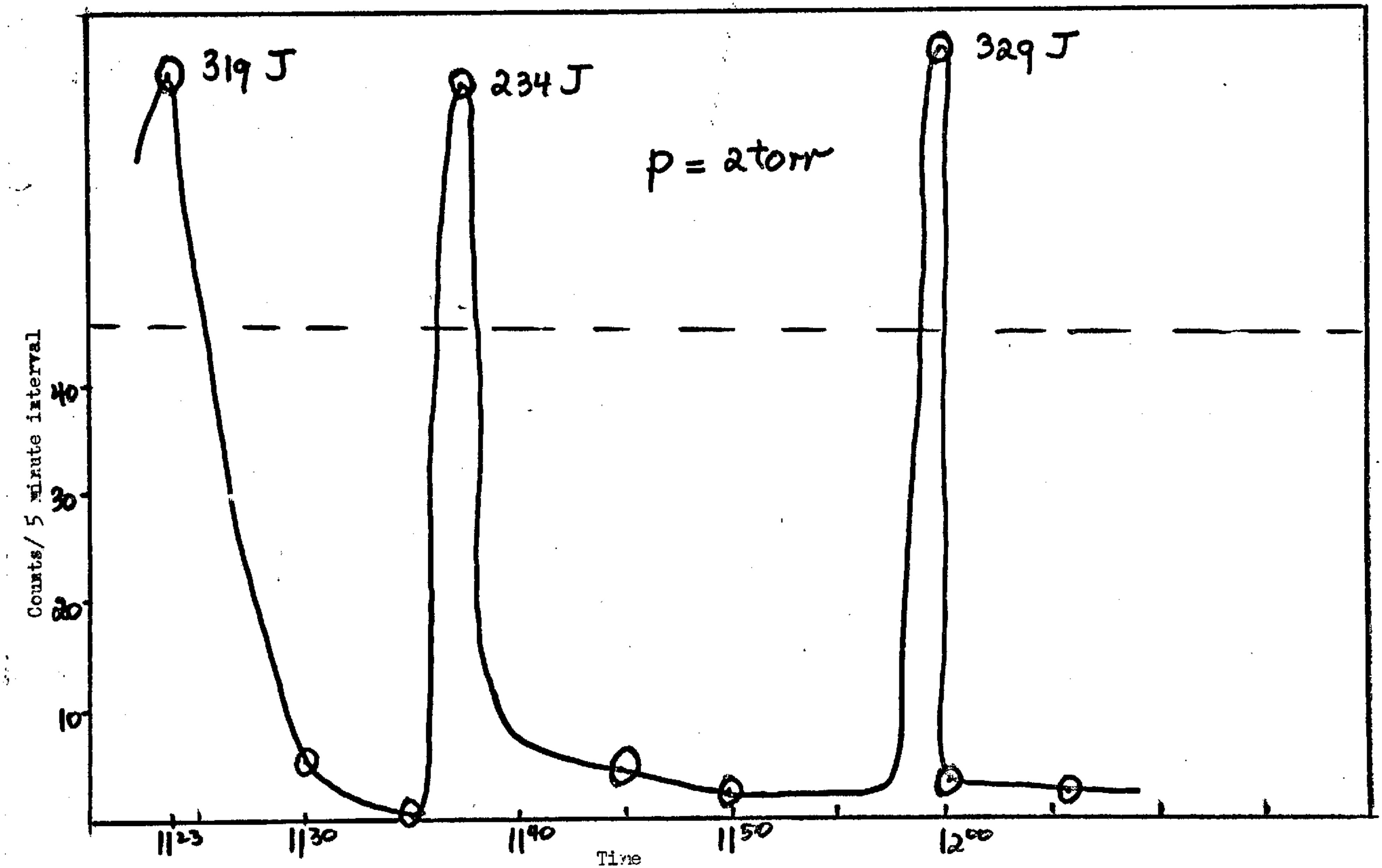

Figure 9b: August 27,74: Series 600 Reynolds connector, after 5 days of continuous outgasing, filled at different pressures with dessicant-dried air

(1/3)

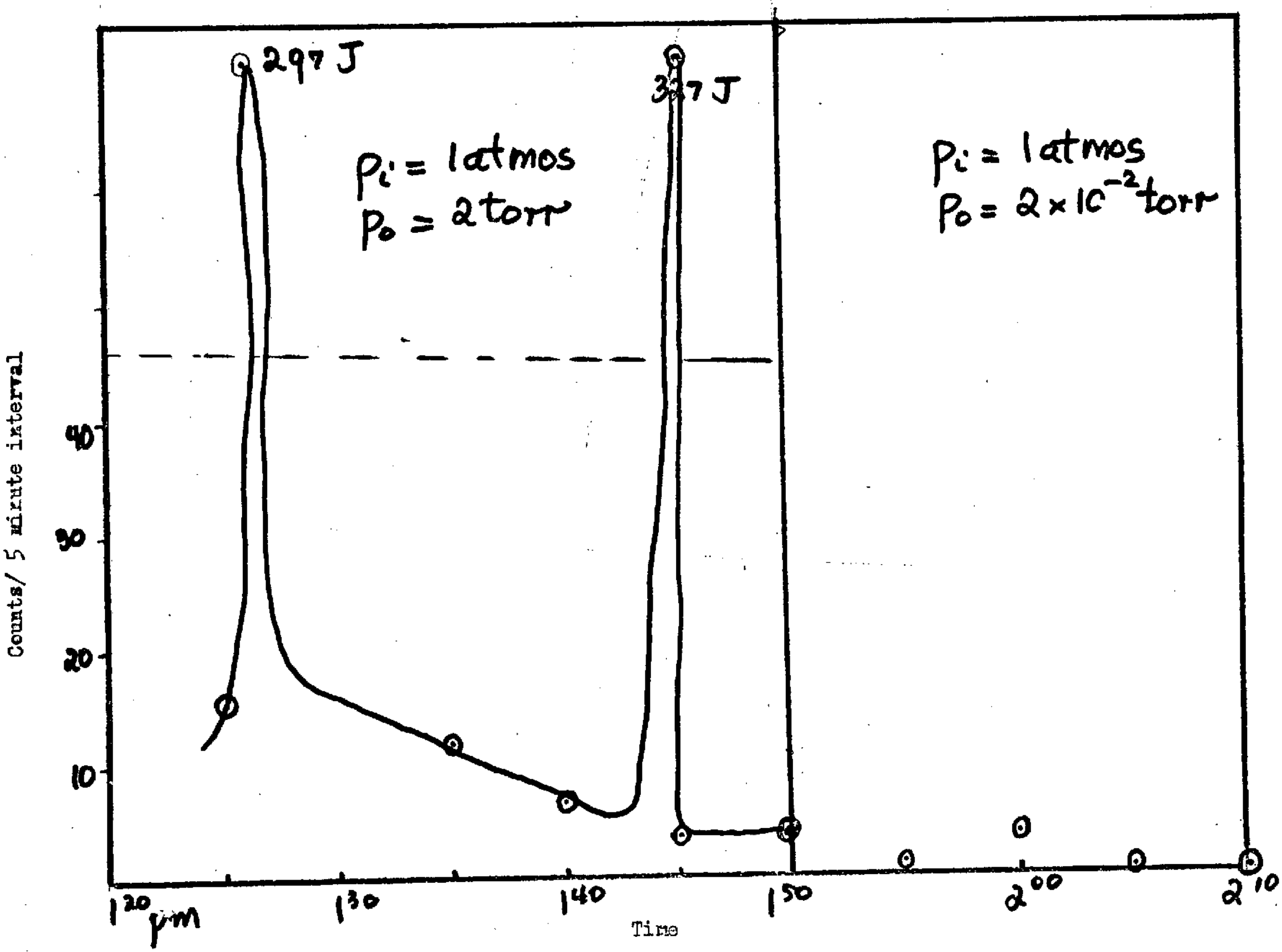

Figure 9c: August 27, 74: Series 600 Reynolds connector, after 5 days of continuous outgasing, filled at different pressures with dessicant-dried air

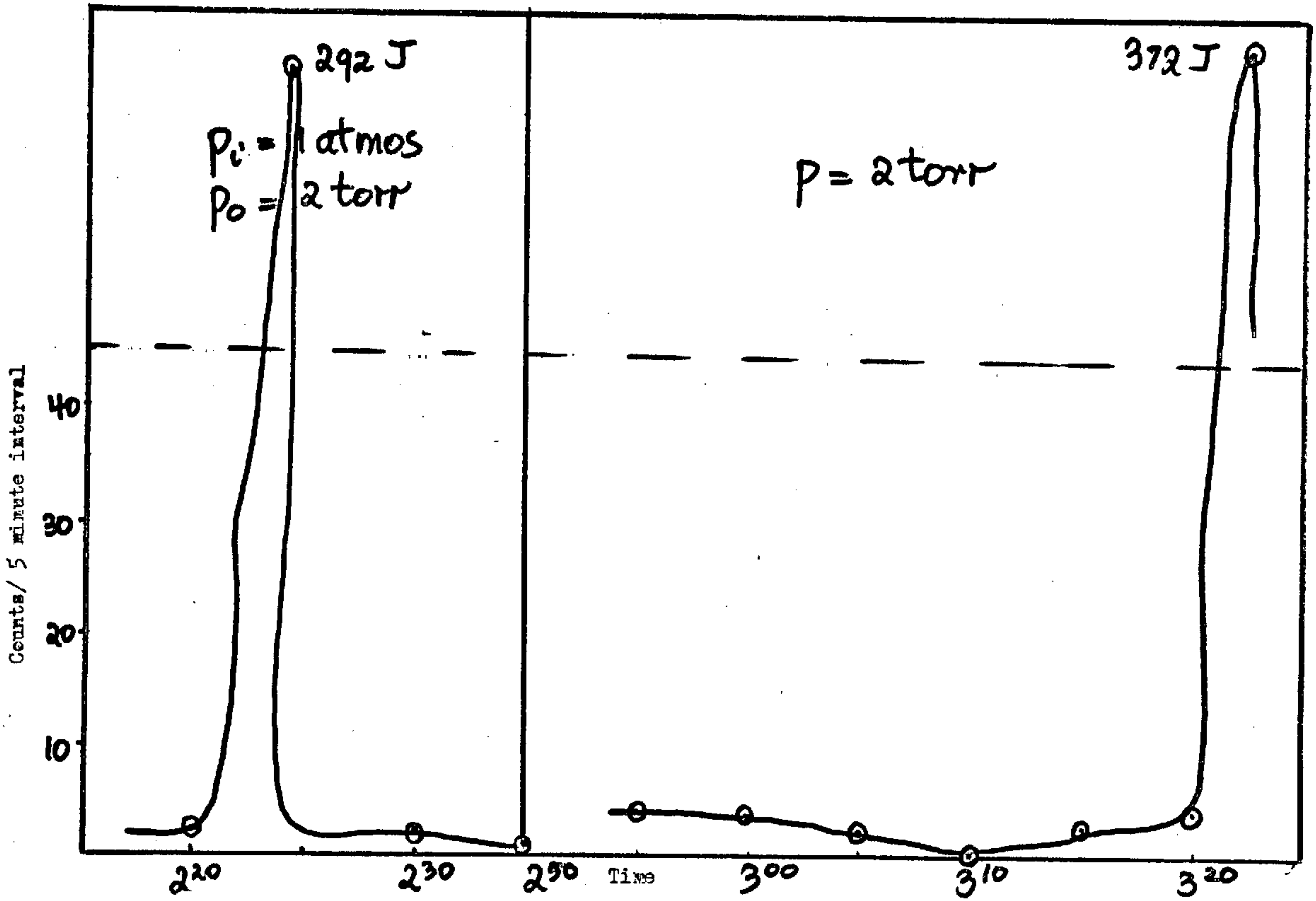

Figure 9d: August 27, 74: Series 600 Reynolds connector, after 5 days of continuous outgasing, filled at different pressures with dessicant-dried air

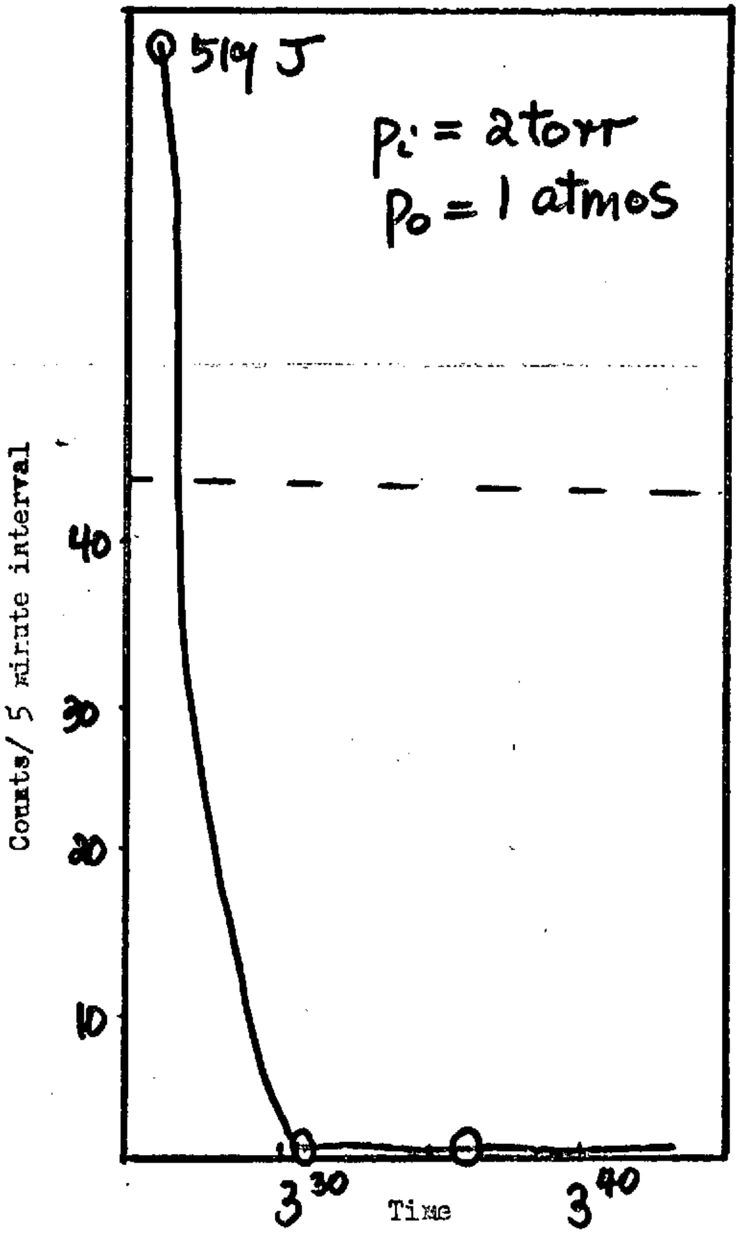

Fig. 9e: August 27,74: Series 600 Reynolds connector, after 5 days outgasing, filled at different pressures with dessicant-dried air

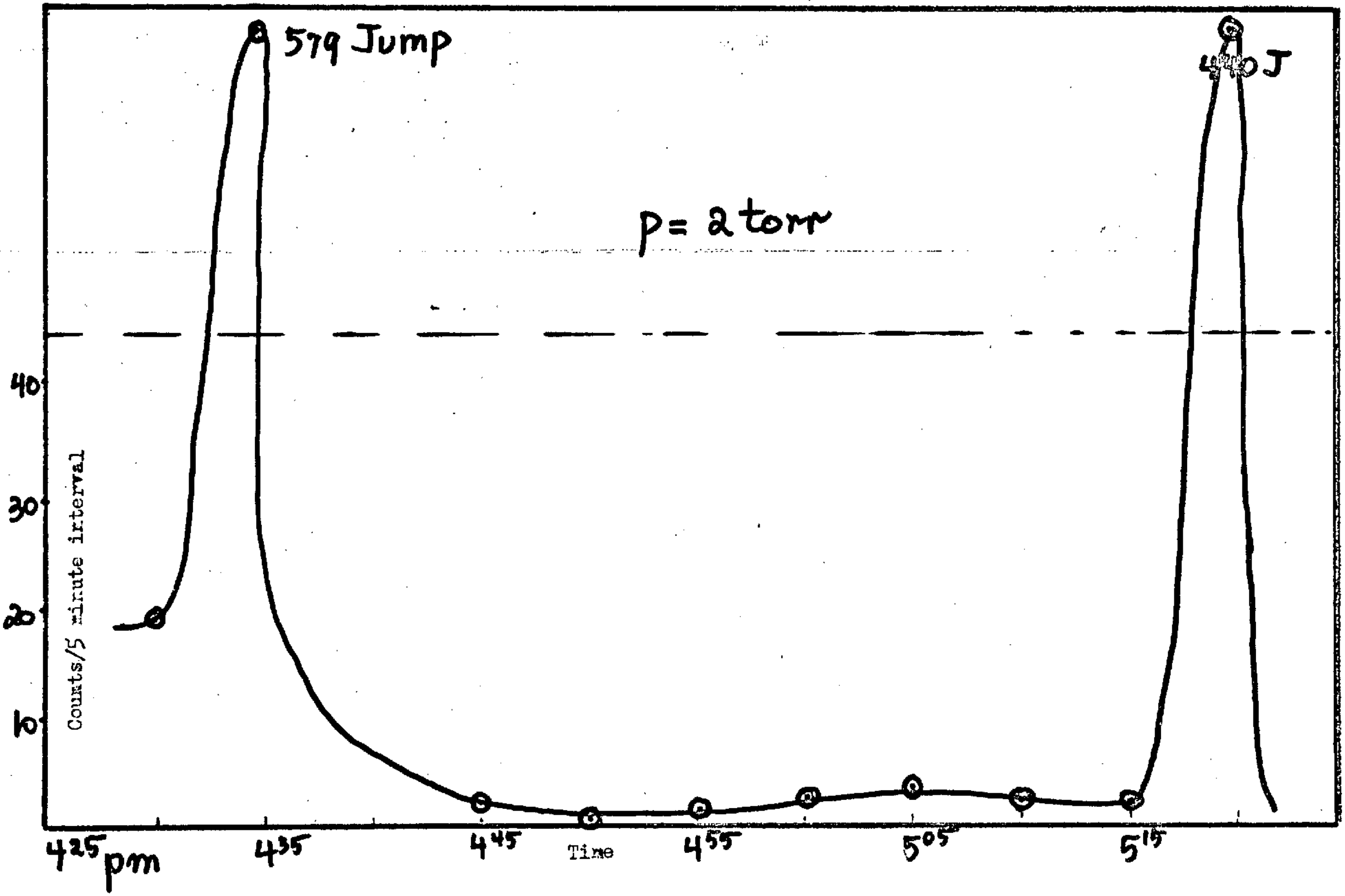

Figure 10 a : August 28,74: Series 600 Reynolds connector, after 5 days of outgasing, filled at different pressures with dessicant-dried air.

Aug 29

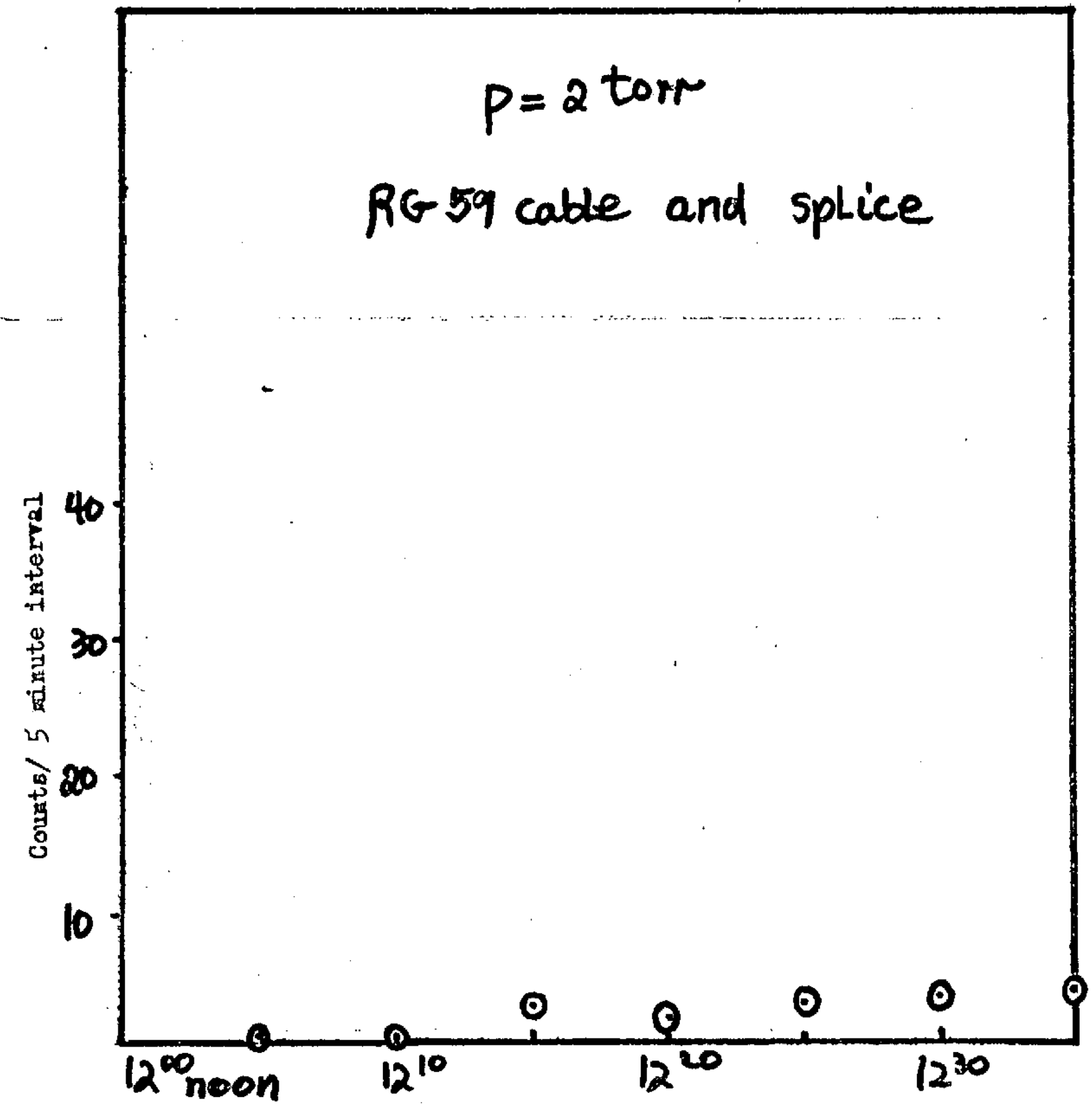

Figure 11: August 29,74: RG 59 cable and splice at 2 torr

Sept 16

1/5

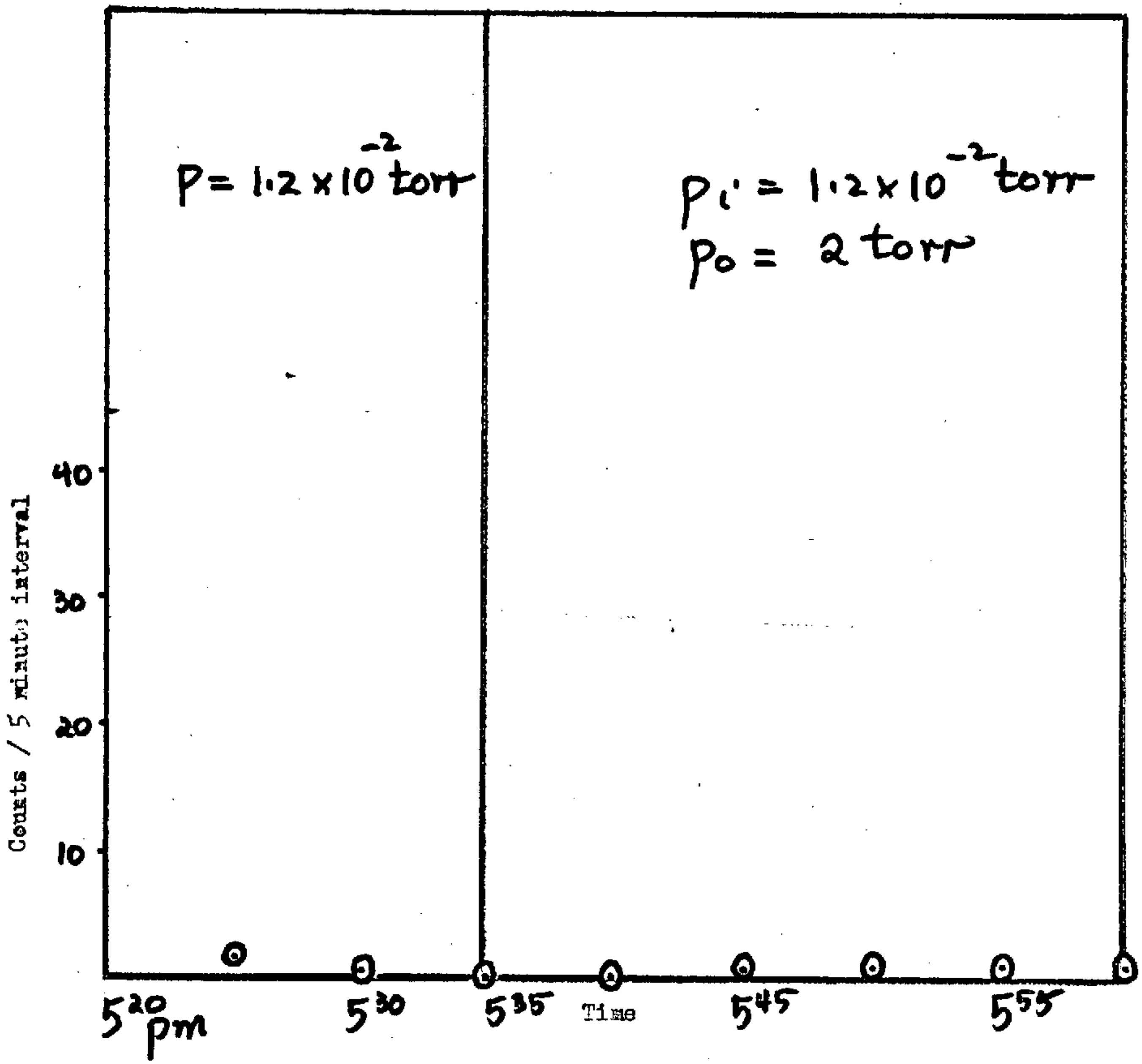

Figure 12 a : Series 600 Reynolds connector.Where cable fits into connector it is potted in Stycast 3050 epoxy. September 16,74. Dry nitrogen used as leak-in gas.

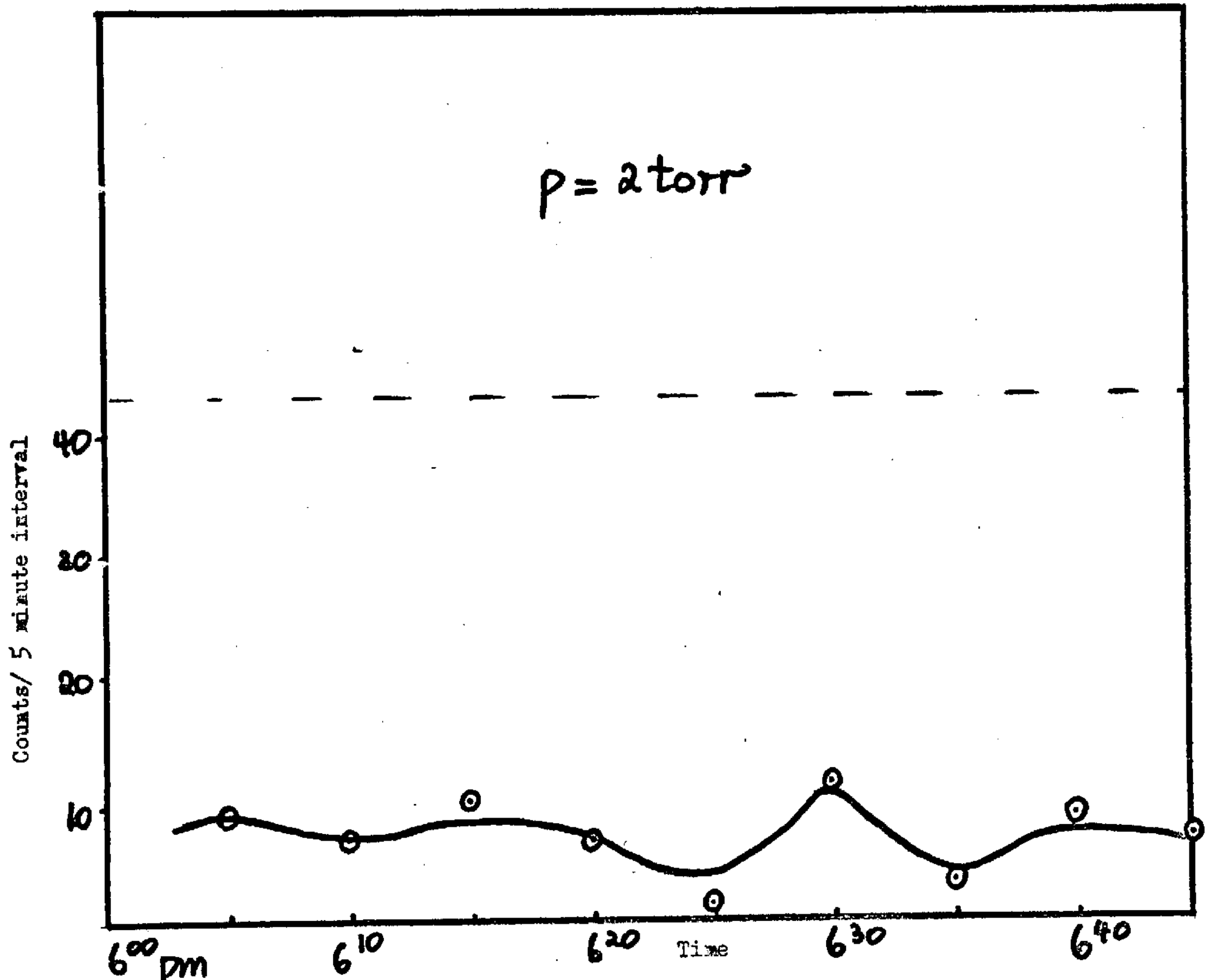

Figure 12 b : Series 600 Reynolds connector. Where cable fits into connector it is potted in Stycast 3050 epoxy. Dry nitrogen used as leak-in gas. September 16, 74

Sept 17

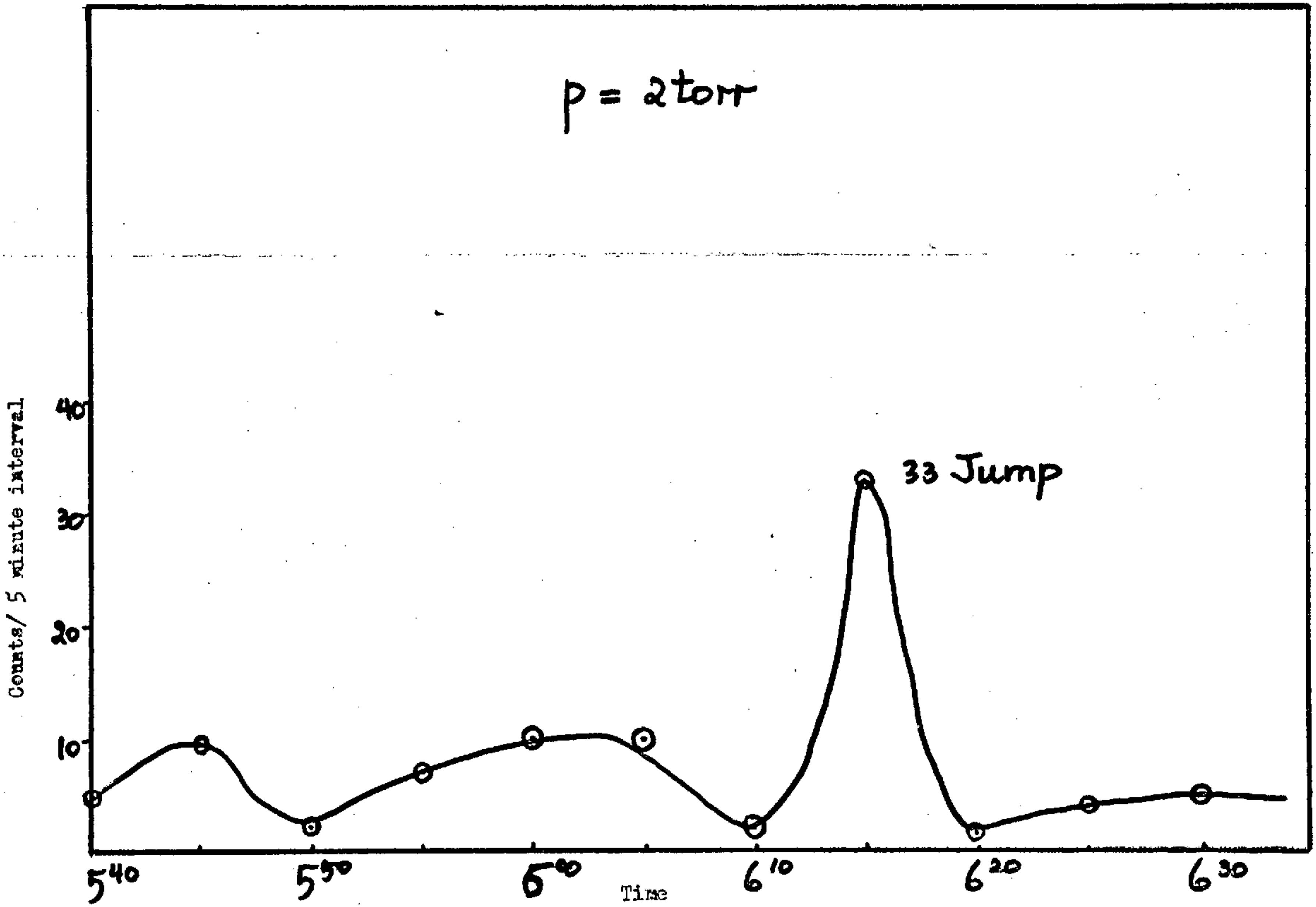

Figure 12 c : Series 600 Reynolds connector. Where cable fits into connector it is potted in Stycast 3050 epoxy. Dry nitrogen used as leak-in gas. September 17, 1974.